Sampling QA/QC Work Plan

STANDARD SCRAP METAL

Prepared By:

Ecology & Environment, Inc.

U.S.EPA Project No.: T05-9410-143
Contractor Project No.: EIL0831FAA
U.S.EPA Contract No.: 68-WO-0037

Approvals

Ecology & Envirronment, Inc.

Raghu Nagam 11/30/94
RAGHU NAGAM Date
Task Leader

EPA

STEVE FARYAN Date
On-Scene Coordinator/
Remedial Project Manager

Raghu Nagam 11/30/94
RAGHU NAGAM Date
Project Manager

1.0 SITE BACKGROUND

The site is located in the City of Chicago which is located in Cook County in the State of IL. The nearest residents are located within 10.0 meters to the south.

It is a scrap metal recycling site on 2.7 acres which is still active. The site began operation in 1987. The site has been operating for approximately 7 years.

The following remedial units are present at the site: waste piles, and storage areas.

The following types of materials were handled at the site: inorganics, and polychlorinated biphenyls.

The contaminants of concern are:

Contaminant	Concentration Range
PCB'S	0-1,700 PPM
LEAD	0-30,000 PPM

The volumes of contaminated materials to be addressed are: PCB'S and lead contaminated soils approximating 3,500 cubic yards.

The suspected contamination is a result of: past activities at the site.

The physical/chemical threat to the population at risk is: direct contact and wind blown contaminated particulates with PCB'S and heavy metals.

The following sampling constraints have been identified: the site being still active poses constraints on sampling.

The following additional information is known about the site: the site has been in operation from about 1920. Operations included salvaging metal from motors etc. and shredding metal.

The basis of the site information is: site assessment report for Standard Scrap Metal by ECOLOGY & ENVIRONMENT, INC.

The current stage/phase of the project is: Cleanup Attainment.

2.0 DATA USE OBJECTIVES

The following data quality objectives will be applied to this project:

Program Area	Sampling Objective	Data Type
Removal	Identification of hot spots	S/C
Removal	Verification of cleanup	S/C/D
Removal	Extent of contamination	S/C

The required confidence level is 95% for screening data (S), 95% for confirmatory data (C), and 95% for defenitive (D) data.

The rational for confidence levels less than 95% is: not applicable

The data will be evaluated against Federal Regulatory Levels. The RCRA regulatory limit is 5.0 mg/kg for lead utilizing the Total Characteristic Leachate Procedure, and for total lead criteria, OSWER Directive #9355.4-02 for soil lead cleanup level of 500 ppm will be used. PCBs will be evaluated against Toxic Substance Control Act (TSCA) clean-up criteria of 10 ppm.

3.0 SAMPLING DESIGN

The following remedial units will be sampled as indicated.

Remedial Unit	Program Area Sampling Objective	Matrix	Parameter
Storage areas	Removal/Extent of contamination	Soil	Heavy Metals, PCBs
waste pile	Removal/Identification of hot spots	Soil	Heavy Metal Content

Sampling Designs:

Storage Areas, Removal/Extent of contamination, Soil, Metals

The Systematic Random sampling approach will be implemented to determine the areal and vertical extent of contamination (EOC). Samples will be collected from the following locations and depths/areas: sample locations based on a 25 feet by 25 feet grid labelled A thru Q, and 1 thru 20 (Sample location grid map). Samples will be collected from 0-1 foot interval, 1-2 foot interval, 2-3 foot interval and 3-4 foot intervals before excavation. The EOC study will also enable to determine the volume of contaminated soil. on site can be estimated. The

beginning of the grid will be the north west corner of the site. Grids will be labelled A thru Q going south and 1 thru 20 going east. Each point on the grid will be field screened/sampled for PCBs and metals. PCB screening will be accomplished by analysis with ENSYS kits or by analysis at E & E warehouse using a gas chromatography. Stock pile samples will be composited as follows: 10 - 15 point compositing will be done.

Waste Pile, Removal/Identification of hot spots, Soil, Heavy Metal Content and PCBs

The Systematic Grid sampling approach will be implemented. Confirmation samples will be collected from the following locations and depths/areas: AT 25 FEET GRID NODES.

Samples will be composited as follows:
After excavation, confirmation sampling will be done by taking five samples within each grid (one from each corner and one from center) and compositing it for analysis in a commercial laboratory and/or in E & E warehouse. Duplicates of these five individual samples will be kept on site for future analysis if needed.

Background samples will be collected from the following locations if needed: LOCAL BALL PARK ON 55TH STREET.

Table 1, Sampling Summary, identifies the number of field samples and QA/QC samples to be collected.

4.0 SAMPLING AND ANALYSIS

Table 2, Sampling Requirements Summary, contains information pertinent to sampling, such as the sample container types and the quantity of sample to be collected at each sampling location, the preservation method to be used, the sample holding times (based on the parameter being analyzed for and the matrix). For the air matrix, this table identifies the sample flow rate rather than sample containers and the volume to be collected rather than the preservative.

The following sampling equipment media will be used to obtain environmental samples from the respective matrix:

Parameter/Matrix	Equipment/Media	Fabrication	Dedicated
Heavy Metal Content/Soil	Backhoe	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		

Parameter/Matrix	Equipment/Media	Fabrication	Dedicated
Heavy Metal Content/Soil	Scoop	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
Heavy Metal Content/Soil	Shovel	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
Heavy Metal Content/Soil	Bucket Auger	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
Heavy Metal Content/Air	Gillian pumps, High VOLs	glass/filter	Y
	Decontamination Steps 1. Physical removal 2. Potable water rinse 3. Air dry		
PCBs/Soil	Auger	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
PCBs/Soil	Backhoe	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		

Parameter/Matrix	Equipment/Media	Fabrication	Dedicated
PCBs/Soil	Bucket Auger	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
PCBs/Soil	Scoop	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
PCBs/Soil	Shovel	carbon steel	N
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash 3. Potable water rinse 4. Air dry		
PCBs/Air	Gillian Pumps, High VOLs	glass/filter	Y
	Decontamination Steps 1. Physical removal 2. Non-phosphate detergent wash		

Table 3, Analytical Summary, contains the action levels, required detection limits, analytical methods/instrument references, and the associated required data type designation.

5.0 STANDARD OPERATING PROCEDURES

5.1 Sampling SOPs

The following sampling SOPs will be implemented for this project. These are typically applicable procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final project deliverables.

General Field Sampling Guidelines (#2001)

Sampling is the selection of a representative portion of a larger population, universe, or body. Through examination of a sample, the characteristics of the larger body from which the sample was drawn can be inferred. In this manner, sampling can be a valuable tool for determining the presence, type, and extent of contamination by hazardous substances in the environment.

The primary objective of all sampling activities is to characterize a waste site accurately so that its impact on human health and the environment can be properly evaluated. It is only through sampling and analysis that site hazards can be measured and the job of cleanup and restoration can be accomplished effectively with minimal risk. The sampling itself must be conducted so that every sample collected retains its original physical form and chemical composition. In this way, sample integrity is insured, quality assurance standards are maintained, and the sample can accurately represent the larger body of material under investigation.

The extent to which valid inferences can be drawn from a sample depends on the degree to which the sampling effort conforms to the project's objectives. For example, as few as one sample may produce adequate, technically valid data to address the project's objectives. Meeting the project's objectives requires thorough planning of sampling activities, and implementation of the most appropriate sampling and analytical procedures.

Sample Storage, Preservation and Handling (#2003)

Samples should be collected using equipment and procedures appropriate to the matrix, parameters and sampling objective. The volume of the sample collected must be sufficient to perform the analysis requested. Samples must be stored in the proper types of containers and preserved in a manner appropriate to the analysis to be performed.

All samples must be cooled [illegible] from the time of collection until analysis. If a preservative other than cooling is used, the preservative is generally added after the sample is collected, unless the sample container has been pre-preserved by the laboratory. If necessary, the pH must be adjusted to the appropriate level and checked with pH paper in a manner which will not contaminate the sample.

Quality Assurance/Quality Control Samples (#2005)

QA samples are used as an assessment tool to determine if environmental data meet the quality criteria established for a specific application. QC samples are generally used to establish intralaboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. The goal of including QA/QC samples with any sampling or analytical event is to be able to identify, measure and control the sources of error that may be introduced from the time of sample bottle preparation through analysis.

Analytical results for these samples can be used to assess accuracy as well as cross contamination. Accuracy refers to the correctness of the concentration value and the qualitative certainty that the analyte is present. It is a combination of both bias (systematic error) and precision (random error). Bias is defined as the deviation of a measured value from a reference value or known spiked amount, and is determined by calculating percent recovery. Precision is a measure of the closeness of agreement among individual measurements. Precision is determined by coefficient of variation calculations.

Sampling Equipment Decontamination (#2006)

Removing or neutralizing contaminants from equipment minimizes the likelihood of sample cross contamination, reduces or eliminates transfer of contaminants to clean areas, and prevents the mixing of incompatible substances. The first step, a soap and water wash, removes all visible particulate matter and residual oils and grease. This may be preceded by a steam or high pressure water wash to facilitate residuals removal. The second step involves a tap water rinse and a distilled/deionized water rinse to remove the detergent. An acid rinse provides a low pH media for trace metals removal and is included in the decontamination process if metal samples are to be collected. It is followed by another distilled/deionized water rinse. If sample analysis does not include metals, the acid rinse step can be omitted. Next, a high purity solvent rinse is performed for trace organics removal if organics are a concern at the site. Typical solvents used for removal of organic contaminants include acetone, hexane, or water. Acetone is typically chosen because it is an excellent solvent, miscible in water, and not a target analyte on the Priority Pollutant List. If acetone is known to be a contaminant of concern at a given site or if Target Compound List analysis (which includes acetone) is to be performed, another solvent may be substituted. The solvent must be allowed to evaporate completely and then a final

distilled/deionized water rinse is performed. This rinse removes any residual traces of the solvent.

Soil Sampling (#2012)

Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel,or scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or, if required, a backhoe.

Waste Pile Sampling (#2017)

Stainless steel shovels, trowels, or scoops should be used to clear away surface material before samples are collected. For depth samples, a decontaminated auger may be required to advance the hole, then another decontaminated auger used for sample collection. For a sample core, thin-wall tube samplers or grain samplers may be used. Near surfaces, samples can be collected with a clean stainless steel spoon or trowel.

All samples collected, except those for volatile organic analysis, should be placed in a Teflon lined or stainless steel pail and mixed thoroughly before transfer to appropriate sample containers.

5.2 Sample Documentation

All sample documents will be completed legibly and in ink. Any corrections or revisions will be made by lining through the original entry and initialling the change. The following sample documentation will be maintained:

Field Logbook

The field logbook is a descriptive notebook detailing site activities and observations so that an accurate, factual account of field procedures may be reconstructed. All entries will be signed by the individuals making them. Entries should include at least the following:

- o site name and project number
- o names of personnel on site
- o dates and times of all entries
- o descriptions of all site activities, including site entry and exit times

- o noteworthy events and discussions
- o weather conditions
- o site observations
- o identification and description of samples and locations
- o subcontractor information and names of on-site personnel
- o dates and times of sample collections and chain of custody information
- o records of photographs
- o site sketches

Field Data Sheets and Sample Labels

Field data sheets and corresponding sample labels are used to identify samples and document field sampling conditions and activities. Field data sheets should be completed at the time of sample collection and should include the following information:

- o site name
- o samplers
- o sample location and sample number
- o date and time the sample was collected
- o type of sample collected
- o brief description of the site
- o weather parameters
- o analyses to be performed
- o sample container, preservation, and storage information

Sample labels will be securely affixed to the sample container. They will clearly identify the particular sample, and should include the following information:

- o site name and project number
- o date and time the sample was collected
- o sample preservation method
- o analysis requested
- o sampling location

Chain of Custody Record

A Chain of Custody Record will be maintained from the time of sample collection until final deposition. Every transfer of custody will be noted and signed for and a copy of the record will be kept be each individual who has signed it. The Chain of Custody Record should include at least the following information:

- o sample identification
- o sample location
- o sample collection date
- o sample information, i.e., matrix, number of bottles collected, etc.
- o names and signatures of samplers

- signatures of all individuals who have had custody of the samples

When samples are not under direct control of the individual currently responsible for them, they will be stored in a locked container which has been sealed with a Custody Seal.

Custody Seal

Custody Seals demonstrate that a sample container has not been opened or tampered with. The individual who has custody of the samples will sign and date the seal and affix it to the container in such a manner that it cannot be opened without breaking the seal.

5.3 Sample Handling and Shipment

Each of the sample bottles will be sealed and caps will be secured with custody seals. Sample bottles will be labeled as described above. Sealed bottles will be placed in the appropriate transport containers and the containers will be packed with an appropriate absorbent material such as vermiculite. All sample documents will be affixed to the underside of each transport container lid. The lid will be sealed and custody seals will be affixed to the transport container.

Regulations for packaging, marking/labeling, and shipping of hazardous materials and wastes are promulgated by the U.S. Department of Transportation (U.S. DOT). Air carriers that transport hazardous materials [illegible] in accordance with the current edition of the International Air Transport Association (IATA) Dangerous Goods Regulations, which applies to shipment and transportation of hazardous materials by air carrier. Following current IATA regulations will ensure compliance with U.S. DOT.

6.0 QUALITY ASSURANCE REQUIREMENTS

The following QA requirements will be implemented on this project:

Screening Data

Screening data is data generated by rapid, non-rigorous methods of analysis, such as test kits and direct-reading instruments. Data indicate presence of compound or class of compounds at an imprecise concentration. Data do not provide definitive analyte identification or quantitation. Data are indicated by color changes or dial readings and

are documented in field logbooks or on field sample data sheets. Examples: GC analysis for PCBs; SPECTRACE XRF - screening for lead.

QA Deliverables for Screening Data

Sample Documentation
Instrument Calibration
Detection Limits

Confirmatory Data

Confirmatory data is data generated by rigorous analytical methods, such as CLP methods. Data are analyte-specific, with confirmation of analyte identity and/or concentration.

Instruments produce "raw data" such as chromatograms. Calibration data for field instruments are available. Instrumental analysis confirms both identity and quantitation and generates data sufficient to undergo validation by National Functional Guidelines. Data are found in field instrument printouts and laboratory data packages. Data may be generated in field or "fixed" laboratories as long as QA/QC requirements are met. Confirmatory data is a subset of a larger data set generated by less rigorous methods. The rigorous data set confirms presence and concentrations of compounds detected by less stringent methods. Examples: AA or ICP data under EPA-approved methods; field GC with confirmation by laboratory GC/MS under EPA approved methods.

QA Deliverables for Confirmatory Data

Sample Documentation
Chain of Custody Records
Initial and Continuing Instrument Calibration
Detection Limits
Documentation of Sample Quantitation
Method Blanks, Trip Blanks, Rinsate Blanks
Matrix Spikes or Duplicates
Performance Evaluation (PE) Samples (optional)

Definitive Data

Data are definitive when analytical error is determined. Precision, accuracy, and coefficient of variation are determined for all samples. Error determination may be accomplished through the analysis of eight replicate samples. The same rigorous analytical methods which generate confirmatory data also generate definitive data, providing confirmed analyte identity and quantitation with

additional measures taken to provide error determination. Data are documented in laboratory data packages. Example: GC/MS analysis under EPA-approved methods with PE samples and eight replicate analyses.

QA Deliverables for Definitive Data

Sample documentation
Chain of Custody Records
Initial and Continuing Instrument Calibration
Detection Limits
Documentation of Sample Quantitation
Method Blanks, Trip Blanks, Rinsate Blanks
Matrix Spikes or Duplicates
Performance Evaluation (PE) Samples (required)
Analytical Error (precision, accuracy, coefficient of variation)

7.0 DATA VALIDATION

Data generated for this project will be validated as follows:

Screening Data

Screening data need only be evaluated for calibration and detection limits.

Confirmatory Data

Data generated under this QA/QC Sampling Plan will be evaluated accordingly with appropriate criteria contained in the Removal Program Data Validation Procedures which accompany OSWER Directive #9360.4-1.

The results of 10% of the samples in the analytical data packages should be evaluated for all of the elements listed in Section 6.0 of the QA/QC Sampling Plan. The holding times, blank contamination, and detection capability will be reviewed for all remaining samples.

Definitive Data

Data generated under this QA/QC Sampling Plan will be evaluated accordingly with appropriate criteria contained in Removal Program Data Validation Procedures which accompany OSWER Directive #9360.4-1.

This objective, the most stringent of all objectives, requires that at least 10% of the samples in the lab data

package be evaluated for all of the elements listed in Section 6.0 of this QA/QC Sampling Plan. Of the remaining samples, holding times, blank contamination, precision, accuracy, error determination, detection limits, and confirmed identification will be reviewed. This objective also requires review of all elements for all samples in each analyte category (i.e. VOA's and PCB's) in every tenth data package received from an individual lab.

8.0 DELIVERABLES

The Ecology & Environment, Inc. Task Leader/Manager, RAGHU NAGAM, will maintain contact with the EPA On-Scene Coordinator/Remedial Project Manager, STEVE FARYAN, to provide information regarding the technical and financial progress of this project. This communication will begin when the project is assigned. Activities under this project will be documented and reported in the deliverables described below.

Analytical Report

An analytical report will be prepared for samples analyzed under this plan. Information regarding the analytical methods or procedures employed, sample results, QA/QC results, chain of custody documentation, laboratory correspondence, and raw data will be provided within this deliverable.

Data Review

A review of the data generated under this plan will be undertaken. The assessment of data acceptability or useability will be provided separately, or as part of the analytical report.

Final Report

A (draft) final report will be prepared to correlate available background information with data generated under this sampling event and identify supportable conclusions and recommendations which satisfy the objectives of this sampling QA/QC plan.

Maps/Figures

The following illustrations will be provided:

Maps

Figures
Drawings

9.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

9.1 Personnel Information

The EPA On-Scene Coordinator/Remedial Project Manager, STEVE FARYAN, will provide overall direction to the Ecology & Environment, Inc. staff concerning project objectives, sampling needs, and schedule.

The Ecology & Environment, Inc. Task Leader/Manager, RAGHU NAGAM, is the primary point of contact with the EPA On-Scene Coordinator/Remedial Project Manager. The Task Leader/Manager is responsible for the development and completion of the Sampling QA/QC Plan, project team organization, and supervision of all project tasks.

The Ecology & Environment, Inc. Site QC Coordinator, Dave Hendrin, is responsible for ensuring field adherence to the Sampling QA/QC Plan and recording any deviations. The Site QC Coordinator is also the primary contact with the analytical laboratory.

The following personnel will also work on this project:

Name	Responsibility
Dave Hendrin	QC coordinator
Mary Jane Ripp	QA/QC Coordinator

9.2 Laboratory Information

The following laboratories will be providing the following analyses:

Lab Name/Location	Lab Type	Parameters
EMT/Morton Grove, IL		Total & TCLP metals, PCBs
QAL/Lisle, IL		Total & TCLP metals, PCBs
NATLSCO/Long Grove,IL		Lead and PCBS

10.0 SCHEDULE OF ACTIVITIES

Proposed Schedule of Work

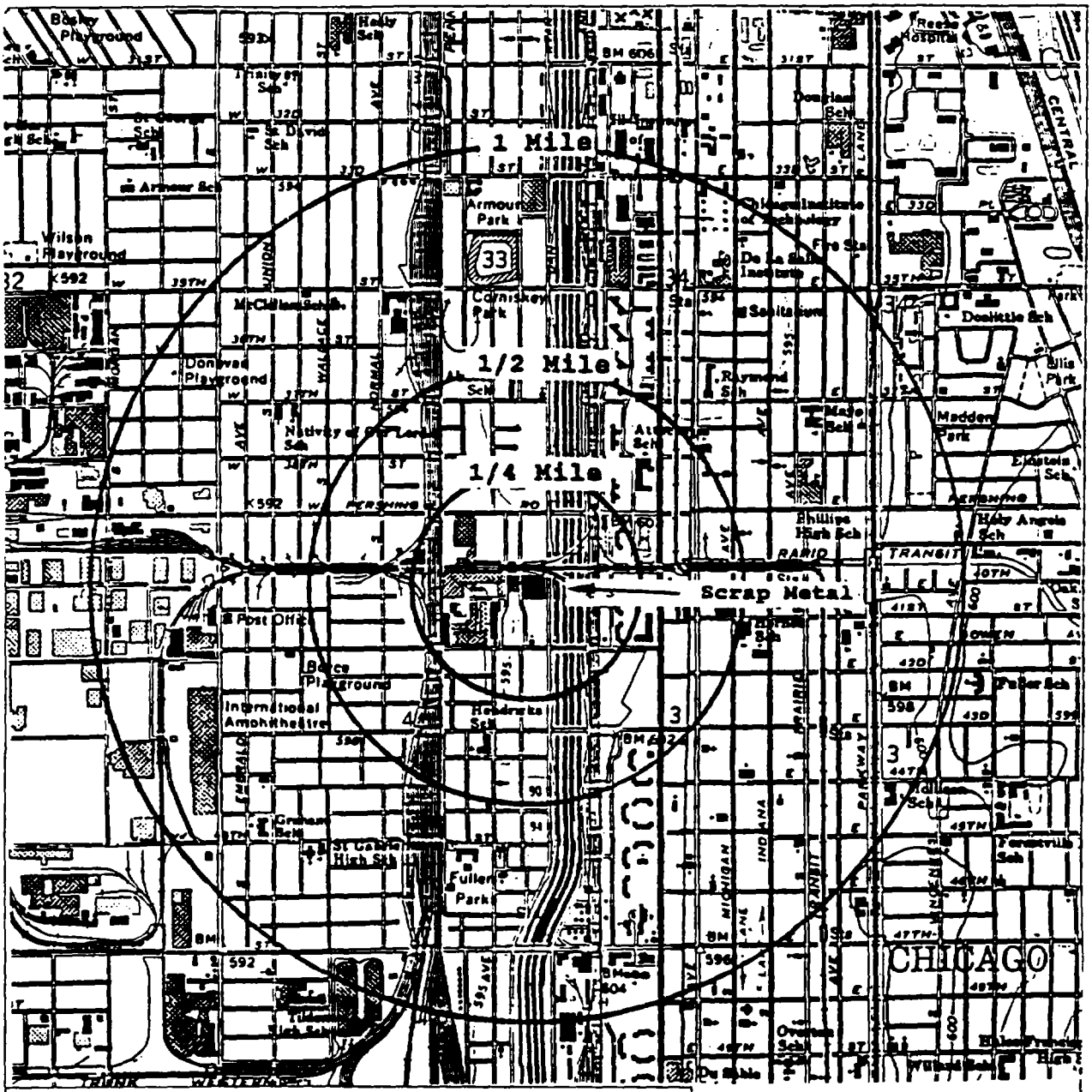
Activity	Start Date	End Date
sampling	11/01/94	02/25/95
mobilization	11/01/94	11/05/94
demobilization	05/25/95	05/30/95

11.0 ATTACHMENTS

The following are attachments to this Sampling QA/QC Plan:

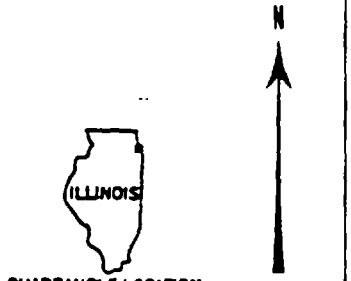
- Site Location Map
- Grid Sample Location Map
- E & E SOP's
- XRF - SOP's
- ENSYS - SOP's
- Target Analyte List - Inorganics
- Target Compound List - Pesticides/PCBs

Standard Scrap
Figure 1 - Site Location Map

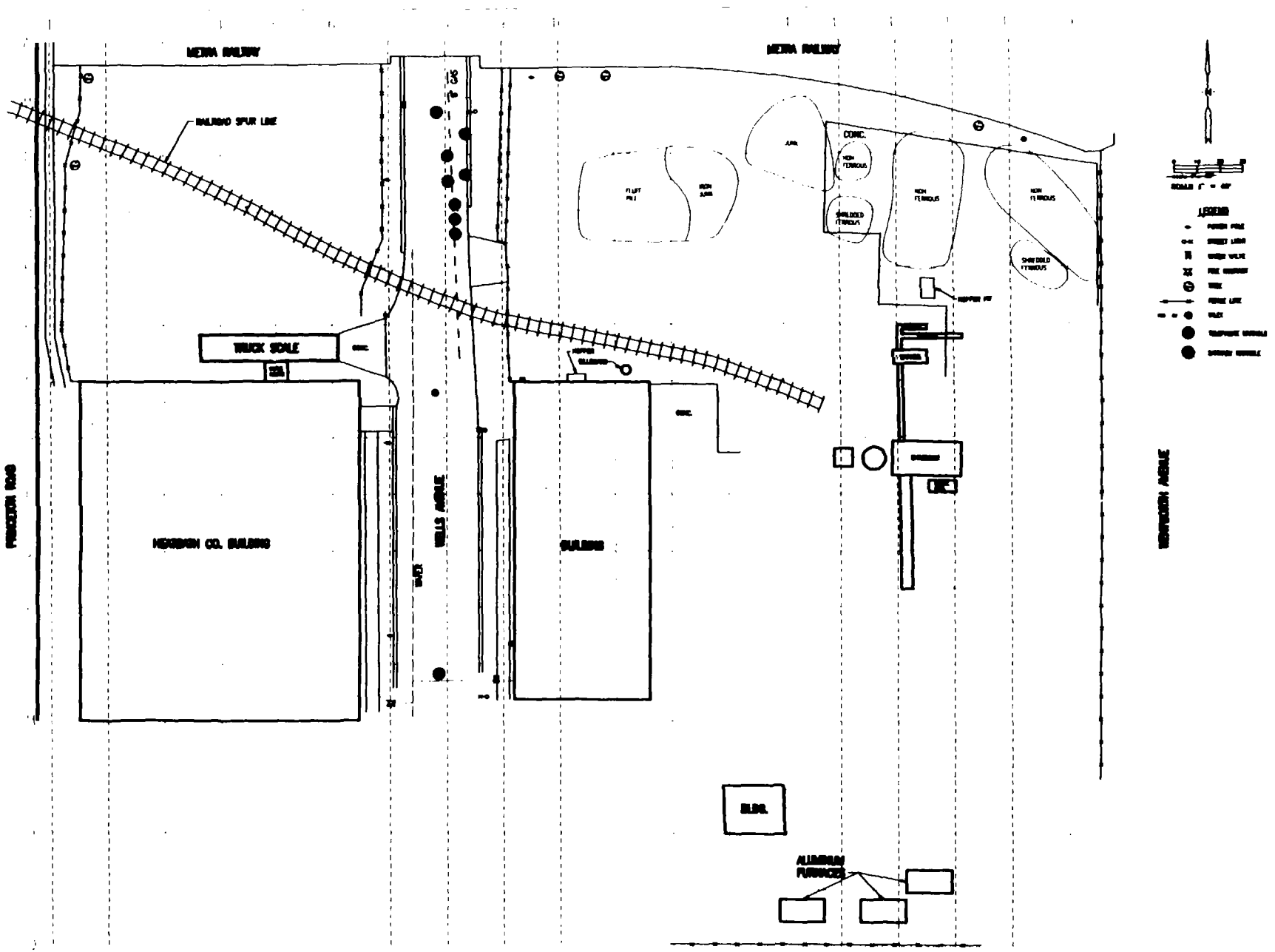


ecology and environment, inc.
Technical Assistance Team
Region V
111 W. Jackson Blvd., Chicago, Illinois 60604

TITLE: SITE LOCATION MAP	FIGURE #: 1
SITE: SCRAP METAL	SCALE: 1:24,000
CITY: CHICAGO STATE: ILLINOIS	TDD: TO5-9311-007
SOURCE: USGS TOPO ENGLEWOOD, IL, JACKSON PARK, IL	DATE REVISED: 1980, 1972



Standard Scrap
Figure 2 - Grid sample location map



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REVISIONS

TOPOGRAPHY AND SAMPLE LOCATION MAP

INORGANIC TARGET ANALYTE LIST (TAL)

Analyte	Detection Limit (ug/L -- water (1))
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

(1) Sediment detection limit 100x water (ug/kg soil/sediment).

Based on the Contract Laboratory Program Statement of Work, ILM02.1 (9/91).

TARGET COMPOUND LIST (TCL) AND
QUANTITATION LIMITS (QL) (1)

	Pesticides/PCBs	CAS Number	Quantitation Limits(2) Water Low ug/L	Soil/Sediment(3) ug/Kg
98.	alpha-BHC	319-84-6	0.05	1.7
99.	beta-BHC	319-85-7	0.05	1.7
100.	delta-BHC	319-86-8	0.05	1.7
101.	gamma-BHC (Lindane)	58-89-9	0.05	1.7
102.	Heptaclor	76-44-8	0.05	1.7
103.	Aldrin	309-00-2	0.05	1.7
104.	Heptachlor epoxide	1024-57-3	0.05	1.7
105.	Endosulfan I	959-98-8	0.05	1.7
106.	Dieldrin	60-57-1	0.10	3.3
107.	4,4'-DDE	72-55-9	0.10	3.3
108.	Endrin	72-20-8	0.10	3.3
109.	Endosulfan II	33213-65-9	0.10	3.3
110.	4,4'-DDD	72-54-8	0.10	3.3
111.	Endosulfan sulfate	1031-07-8	0.10	3.3
112.	4,4'-DDT	50-29-3	0.10	3.3
113.	Methoxychlor	72-43-5	0.50	17.0
114.	Endrin ketone	53494-70-5	0.10	3.3
115.	Endrin aldehyde	7421-36-3	0.10	3.3
116.	alpha-Chlordane	5103-71-9	0.5	1.7
117.	gamma-Chlordane	5103-74-2	0.5	1.7
118.	Toxaphene	8001-35-2	1.0	170.0
119.	Aroclor-1016	12674-11-2	0.5	33.0
120.	Aroclor-1221	11104-28-2	0.5	33.0
121.	Aroclor-1232	11141-16-5	0.5	67.0
122.	Aroclor-1242	53469-21-9	0.5	33.0
123.	Aroclor-1248	12672-29-6	0.5	33.0
124.	Aroclor-1254	11097-69-1	1.0	33.0
125.	Aroclor-1260	11096-82-5	1.0	33.0

(1) Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

(2) Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment on dry weight basis will be higher.

(3) Medium Soil/Sediment Quantitation Limits (QL) for Pesticides/PCB TCL compounds are 15 times the individual Low Soil/Sediment QL.

Based on the Contract Laboratory Program Statement of Work, OLM01.6 (6/91).

Table 1. Field Sampling Summary

Remedial Unit: waste pile
Date: 11/02/94
Program Area: Removal
Page: 1
Sampling Objective: Identification of hot spots

Matrix	Parameter	Backgrnd Samples	Screening Samples	Confirm Samples	Trip Blanks	Field Blanks	Rinsate Blanks	Sample Replicates	PE Samples	Total Samples
Soil	Heavy Metal Content	1	10	1	1	1	1	0	0	15
Soil	Pesticides/PCBs	0	0	10	1	1	1	0	0	13

Table 1. Field Sampling Summary

Remedial Unit: storage areas
Program Area: Removal
Sampling Objective: Extent of contamination

Date: 11/02/94
Page: 2

Matrix	Parameter	Backgrnd Samples	Screening Samples	Confirm Samples	Trip Blanks	Field Blanks	Rinsate Blanks	Sample Replicates	PE Samples	Total Samples
Soil	Metals	0	0	300	30	30	30	0	0	390

Table 2. Sampling Requirements Summary

Date: 11/02/94
Page: 1

Remedial Unit	Program Area/Sampling Object	Matrix	Parameter	Sample Container (Number)	Sample Preservation	Sample Holding Time
storage areas	Removal/Extent of contamination	Soil	Metals	4 oz glass bottle	NA	6 months
waste pile	Removal/Identification of hot spots	Soil	Heavy Metal Content	4 oz glass bottle	NA	6 months
waste pile	Removal/Identification of hot spots	Soil	Pesticides/PCBs	4 oz glass bottle	NA	7/40 days
Site Air	Monitoring/Personal Exposure	Air	PCBs/lead	Hopcolite Tube Filter	NA	7/60 days

Table 3. Analytical Summary

Date: 11/02/94
Page: 1

Remedial Unit	Program Area/Sampling Object	Matrix	Parameter	Action Level	Required Detection Limit	Analytical Method/ Instrument	Required Data Type
storage areas	Removal/Extent of contamination	Soil	Metals	10 ppm	See attached	SW-846, Method 8080	S/C
waste pile	Removal/Identification of hot spots	Soil	Heavy Metal Content	500 PPM, 10 PPM	Analyte specific	Gas Chromatography , SW-846, METHOD 6010	S/C
waste pile	Removal/Identification of hot spots	Soil	Pesticides/PCBs	10 PPM	See attached	SW-846, Method 8080	S/C
Site	Monitoring/Personal Exposure	Air	Metals PCBs	10 mg/m3 0.05 mg/m3	Analyte Specif.	OSHA ID 121 or Equiv. NIOSH 5503 or Equiv.	

DATA QUALITY OBJECTIVES SUMMARY (Soil and Water Matrices)

Remedial Unit: waste pile
Program Area/Sampling Objective: Removal/Identification of hot spots
Required Data Quality Type: S/C
Parameter: Pesticides/PCBs Matrix: Soil

Sampling Design

Approach: random Number of Samples: 10
Sampling Location, Depths and Areas: at grid nodes of 5 feet intervals
Compositing Scheme: 10 - 15 point compositing will be done
Number of Background Samples: 0
Background Sampling Locations: local

Sampling and Analytical Information

Sample Container Type, Volume(number per location): 4 oz glass bottle
Sample Preservation: NA Sample Holding Time: 7/40 days
Analytical Method/Instrument: SW-846, Method 8080 (C)
Action Level: 10 PPM (C) Detection Limit: See attached
Analytical Error Calculations Required? N
Number of Trip Blanks: 1 Number of Rinsate Blanks: 1
Number of Field Blanks: 1 Number of PE Samples: 0
Number of Replicates/Matrix Spikes: 0

QA Deliverables for Screening Data

1. Sample Documentation
2. Instrument Calibration Records
3. Detection Limits Records

QA Deliverables for Confirmatory Data

1. Sample Documentation, including Chain of Custody Records
2. Initial and Continuing Instrument Calibration Records
3. Detection Limit Records
4. Documentation of Sample Quantitation
5. Documentation of Results of QA//QA Sample: Method Blanks, Trip Blanks, Rinsate Blanks
6. Documentation of Results of Matrix Spikes or Duplicates
7. Documentation of PE Samples (optional)

DATA QUALITY OBJECTIVES SUMMARY (Soil and Water Matrices)

Remedial Unit: waste pile
Program Area/Sampling Objective: Removal/Identification of hot spots
Required Data Quality Type: S/C
Parameter: Heavy Metal Content Matrix: Soil

Sampling Design

Approach: Systematic Grid Number of Samples: 10
Sampling Location, Depths and Areas: GRID NODES POINT AND AT 1 FOOT INTERVALS.
Compositing Scheme:
Number of Background Samples: 1
Background Sampling Locations: LOCAL BALL PARK ON 55TH STREET.

Sampling and Analytical Information

Sample Container Type, Volume(number per location): 4 oz glass bottle
Sample Preservation: NA Sample Holding Time: 6 months
Analytical Method/Instrument: Gas Chromatography (S), SW-846, METHOD 6010 (C)
Action Level: 500 PPM (S), 10 PPM (C) Detection Limit: Analyte specific
Analytical Error Calculations Required? N
Number of Trip Blanks: 1 Number of Rinsate Blanks: 1
Number of Field Blanks: 1 Number of PE Samples: 0
Number of Replicates/Matrix Spikes: 0

QA Deliverables for Screening Data

1. Sample Documentation
2. Instrument Calibration Records
3. Detection Limits Records

QA Deliverables for Confirmatory Data

1. Sample Documentation, including Chain of Custody Records
2. Initial and Continuing Instrument Calibration Records
3. Detection Limit Records
4. Documentation of Sample Quantitation
5. Documentation of Results of QA//QA Sample: Method Blanks, Trip Blanks, Rinsate Blanks
6. Documentation of Results of Matrix Spikes or Duplicates
7. Documentation of PE Samples (optional)

DATA QUALITY OBJECTIVES SUMMARY (Soil and Water Matrices)

Remedial Unit: storage areas
Program Area/Sampling Objective: Removal/Extent of contamination
Required Data Quality Type: S/C
Parameter: Metals Matrix: Soil

Sampling Design

Approach: Syetematic Random Number of Samples: 300
Sampling Location, Depths and Areas: sample locations based on a 25 feet by 25 feet grid (A thru ..., and 1 thru ....) Samples will be collected from 0 - 1 foot interval, 1 - 2 feet interval, and 2 - 3 feet intervals before excavation. After excavation, confirmation sampling will be done by taking five samples within each grid (one from each corner and one from center) and compositing it for analysis in E & E warehouse.
Compositing Scheme: The purpose of this sampling event is to determine the extent of contamination so that the volume of contaminated soil on site can be estimated. The begining of the grid will be the north west corner of the site. Grids will be labelled A thru X going south and 1 thru 15 going east. Each point on the grid will be field screened for PCBs and lead. The Confirmation sampling will be done by collecting five samples from each grid area and compositing them for analysis in E & E warehouse (one sample from each corner and from the center). Duplicates of these five individual samples will be kept on site for future analysis if needed.
Number of Background Samples: 0
Background Sampling Locations: not applicable

Sampling and Analytical Information

Sample Container Type, Volume(number per location): 4 oz glass bottle
Sample Preservation: NA Sample Holding Time: 6 months
Analytical Method/Instrument: SW-846, Method 8080 (C)
Action Level: 10 ppm (C) Detection Limit: See attached
Analytical Error Calculations Required? N
Number of Trip Blanks: 30 Number of Rinsate Blanks: 30
Number of Field Blanks: 30 Number of PE Samples: 0
Number of Replicates/Matrix Spikes: 0

QA Deliverables for Screening Data

1. Sample Documentation
2. Instrument Calibration Records
3. Detection Limits Records

QA Deliverables for Confirmatory Data

1. Sample Documentation, including Chain of Custody Records
2. Initial and Continuing Instrument Calibration Records
3. Detection Limit Records
4. Documentation of Sample Quantitation
5. Documentation of Results of QA//QA Sample: Method Blanks, Trip Blanks, Rinsate Blanks
6. Documentation of Results of Matrix Spikes or Duplicates
7. Documentation of PE Samples (optional)

ecology and environment, inc.

Title: SOP-High Volume Air Sampling

Category: GENTECH 4.10

Revised: September 1987

Approved: R. Gray

STANDARD OPERATING PROCEDURES
FOR
HIGH-VOLUME AIR SAMPLING
AT HAZARDOUS WASTE SITES

Revised: September 1987

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CONTENTS

LIST OF TABLES

1 INTRODUCTION

The Field Investigation Team (FIT) is organized to investigate and evaluate potential contaminant releases from uncontrolled hazardous waste sites for the U.S. Environmental Protection Agency (EPA). To obtain a more complete site characterization or to determine potential health impacts to local inhabitants from possible contaminants in the air, the FIT conducts air monitoring programs. The FIT develops high-volume (hi-vol) air sampling networks, using the hi-vol sampler, a particle filtration instrument that collects total suspended particulates (TSP). TSP determinations involve gravimetric analysis to derive the mass concentration of suspended particulate in ug/m3. After gravimetric analysis the filters can be submitted for a complete chemical analysis if desired. The sampling procedures and methodologies used by the FIT will be in accordance with the Code of Federal Regulations, 40 CFR Part 58, Ambient Air Quality Surveillance, July 1, 1986 (see Appendix A) and the EPA Quality Assurance Handbook for Air Pollution Measurement Systems (1985).

2 PURPOSE

This document establishes Standard Operating Procedures for FIT field operations with respect to program development, collection, and handling of hi-vol samples from designated hazardous waste sites. This SOP serves as a reference for field samplers and provides guidance with respect to policies formulated by the Environmental Services Division (ESD) of EPA and the FIT in Region VIII, and it includes a series of appendices describing sampling program development, sampling procedures, and handling of samples.

3 SCOPE

The procedures outlined in this SOP are applicable to all FIT personnel participating in the collection of hi-vol samples from hazardous waste sites. The following sections describe various phases of sampling activity.

4 SAMPLING PROGRAM ORGANIZATION

4.1 COORDINATING INSTRUCTIONS

It is critical that all sampling activities be planned well in advance so that the sampling can be coordinated between the different parties involved. Table 4-1 lists the various steps involved with each sampling activity and the person responsible for coordination. All air-sampling plans must be submitted to ESD at least 3 weeks before field sampling begins.

Table 4-1. Coordination of Sampling Activities

Step	Activity
1.	RPO issues TDD to FIT RPM.
2.	RPM selects FIT PO and discusses initial project requirements with him/her.
3.	FIT PO enters TDD in TDD Master Log, opens TDD Master File.
4.	FIT PO discusses project with EPA PO.
5.	FIT PO develops sample plan.
6.	FIT PO submits sample study plan to RPO for review.
7.	FIT PO makes any necessary changes in sample plan following RPO's review.
8.	FIT PO coordinates with ESD for any required EPA lab support.
9.	RPO assigns any required contractor lab support and a case number.
10.	FIT PO discusses equipment needs with FIT equipment officer.
11.	FIT PO coordinates field preparations, including equipment preparations and travel arrangements.
12.	EPA PO coordinates with governmental and private parties regarding access and scheduling. The FIT PO will usually assist the EPA PO in making landowner contacts to gain permission to install samplers and gain pover. In many cases, local agencies such as the county health department will assist with these contacts.
13.	FIT samples site.
14.	FIT transmits samples to appropriate lab facilities for analysis, in accordance with RPO's instructions.
15.	FIT PO submits sampling activities report, documenting field activities to RPO.
16.	Lab analyzes samples and forvards data to RPO.
17.	RPO transmits data to FIT PO.
18.	RPO issues TDD for development of final interpretive report based on analysis of data.
19.	FIT PO prepares final interpretive report.
20.	FIT PO submits final interpretive report to RPO.
21.	FIT PO completes the following documents: a. Acknovledgment of Completion for TDD b. Contractor Performance Evaluation (in conjunction with RPO) c. Project Expense Form
22.	FIT PO ensures that copies of all sampling-related papervork are present in TDD file (traffic reports, custody forms, lab request, logbook, airbills, etc.).
23.	FIT PO closes out TDD in Master Log.

Legend: FIT--Field Investigation Team
EPA--Environmental Protection Agency
ESD--Environmental Services Division
PO--Project Officer
RPM--Regional Project Manager
RPO--Regional Project Officer
TDD--Technical Directive Document

4.2 SAMPLING STRATEGY

4.2.1 Location of Sites

Hazardous substance sites are normally sampled either to support enforcement actions or to further characterize a site for remedial work. In addition, hi-vol air sampling can be used to determine if the migration of contaminated suspended particulate matter exists and poses an imminent health threat to local populations. Air monitoring programs can also provide data to support contamination-related health studies, from which correlations may be made between observed health effects and observed air quality exposures. Ideally, the strategy involved with these types of sampling efforts requires the location of sampling sites in areas where the prevailing local winds will most likely carry the highest concentration of particulate matter from the site to the hi-vol units. Samplers should be located both upvind and downvind of the sampling site. The difference between the downvind and upvind concentrations gives the contribution of particulates from the site to the overall particulate concentration. This siting is inherently biased, and routine random-siting techniques are not used. Air sampling should not be attempted if there is snov covering the ground or if the soil is saturated from heavy rain.

4.2.2 Number of Samples

The number of samples to be collected is determined by a number of factors, including the amount of time and financial support provided for the study. An optimum design for the purpose of statistical data analysis would include at least 10 sampling days. Ten replicates at each sample site, including background, enables the investigator to meet the assumptions of a student's t-test. A t-test is recommended to compare samples collected at downvind versus background sites. An explanation of the analytical procedure can be found in an introductory statistics text book.

4.3 WORK PLAN DEVELOPMENT

4.3.1 Sampling Plan

A sampling plan describes the purpose and goals of a field investigation and provides details of methodologies and safety procedures to be used.

Because the data generated from analysis of samples often provide a crucial portion of the evidence used in subsequent litigation and may further be used in the development of appropriate remedial action alternatives, the design of the sampling program must ensure that the samples obtained will meet the goals of the investigation. Careful selection of sampling locations and methods also helps reduce the costs of labor and analytical support.

An example of a sampling plan for hi-vol air sampling can be found in Appendix B. Though the sampling plan is a formal document that

should be brief and concise, the exact organization is flexible; but it should always contain the basic elements presented in Table 4-2.

4.3.2 Safety Plan

An unabridged safety plan should always be completed for any hi-vol air sampling program.

4.4 SAMPLING EQUIPMENT LIST

A list of equipment normally required for a hi-vol sampling program is presented in Table 4-3.

4.5 FIELD OPERATIONS

4.5.1 Siting

4.5.1.1 Reconnaissance activities

Before developing a sampling plan, schedule a site reconnaissance trip to facilitate hi-vol siting. If this cannot be accommodated, 1 or 2 days must be scheduled for siting activities as part of, but before, the actual sampling program begins. These activities can become time consuming and sometimes difficult. Hi-vol sampling usually does not take place on a hazardous waste site, but on adjacent property. Wind rose data for the area to be sampled must be obtained from the Regional ESD--Air Quality Assurance (QA) Laboratory, the National Weather Service, or other local weather stations (such as at airports) in the study area. General siting location areas can be determined from the wind direction patterns and a topographic map.

4.5.1.2 Access to electrical pover

Final confirmation of the sampling locations depends on several factors. The most important factor is the availability of electrical power to operate the hi-vols. Portable generators should be used only as a last resort. In some cases, the most desirable sampling locations cannot be used due to lack of access to a pover source. Landowners in the chosen area must be approached, and permission must be requested to erect the hi-vol in a strategic location on their property and to use their electric pover to operate the sampler. The FIT has authorization to offer reasonable compensation to the landowner for any electricity used. Care must be taken not to locate the hi-vols too close to residences because the continuous noise of operation may be annoying. Excessive extension-cord lengths must also be avoided so that electrical resistance in the vire does not become a problem.

4.5.1.3 Security considerations

A secure location is needed to minimize the potential for tampering or vandalism. Fencing may be an appropriate deterrent in situations where children are present. Other factors to consider during siting include future activity on or around the site. Construction or remedial activities may disrupt the sampling procedure and result in an unrepresentative sample.

Table 4-2. Basic Organization of a Sampling Plan

1. Title Page
2. Introduction
3. Objectives
4. Site description
5. Field procedures
   a. Concept of operations
   b. Sampling locations
   c. Coordination
   d. Field safety
   e. Project schedule
   f. Personnel requirements
   g. Control of contaminated materials
6. Logistics
7. Quality control
   a. Sample methods
   b. Chain of custody
8. Sampling report

Table 4-3. Sampling Equipment List

1. High-volume (hi-vol) units--Field Investigation Team (FIT) has acquired some different models; the GMW equipped with a Lirz 310 flow controller, timer assembly, elapsed time indicator, and Dickson Flow recorder are the preferred units for FIT use.
2. Spare hi-vol parts or extra hi-vol units--parts cannibalized from less functional units not presently in field use. These are important and include timers, flow recorders, motors, sampling heads, etc.
3. Angle-iron legs with bolts.
4. Extra motor brushes.
5. Fuses--8 amp.
6. Extra pen cartridges for flow recorder.
7. Flow recorder charts.
8. GMW cartridge filter holder assembly--one per unit plus one extra.
9. Filters.
10. EPA Data Records.
11. EPA envelopes.
12. Clasp envelopes (10 in. by 13 in.).
13. Official Logbook and field forms.
14. Inhalable particulate sampling head--if required.
15. Calibration kit.
    a. Variable resistance or single point orifice calibration unit with faceplate and calibration graph.
    b. Dwyer slack tube water manometer.
    c. Rotometer--optional.
    d. Extra filters for calibration use.
16. Extension cords--FIT has 500-ft cords.
17. Padlocks.
18. Stepladder.
19. Sledgehammers--8 lb.
20. Level.
21. Generator and gasoline cans, if required.
22. Tape--electrical and fiber.
23. Toolkit--vise grips, screwdrivers, hacksaw, electric drill and bits, crescent wrench, needlenose pliers, and snips.
24. Shovel.
25. Fencing and T-posts.
26. Wire.
27. Knife.
28. Flagging tape.
29. Five-gal. water jug.
30. White cotton gloves.
31. Camera and film.
32. Heavy-duty plastic bags (12 in. by 20 in.).
33. Plastic garbage bags.
34. Paper towels.
35. EPA custody seals.

(Continued)

Table 4-3. Sampling Equipment List (Continued)

36. Wrist chronograph.
37. Hand calculator.
38. Thermoluminescent Device badge.
39. Air purifying respirators with dust cartridges.
40. Field clothing--Level D.
41. Meteorological Station
    a. Wood shelter with legs.
    b. Meteorograph with chartpaper, D-cell batteries, and cord.
    c. Wind speed/direction recorder, chart paper, cord, and spare fuses.
    d. Windspeed/direction vane assembly, 1/2-in. pipe, and angle-iron mounting bracket with extension.
    e. Rope and wire.
    f. T-posts--for anchoring shelter.
    g. Compass.
    h. Mercury thermometer.

4.5.1.4 Background samples

In addition to the siting of hi-vol units adjacent to the study area, a unit must be designated to collect background samples. Because it is used for comparison with concentrations in samples from the hazardous waste site, great care is exercised in the selection of the background sampling location. This unit should be located upwind from the contaminant source. The background sample is representative of conditions existing on the site before hazardous waste was deposited. For additional siting criteria and requirements, refer to Appendix A in this report (40 CFR Part 58, Appendix E, "Probe Siting Criteria for Ambient Air Quality Monitoring").

4.5.2 Equipment Preparation

Before deploying any hi-vol unit in the field, the FIT should complete operational and calibration checks to ensure that it is fully functional. The hi-vol should be equipped with a motor/blower, flow controller, elapsed time indicator, programmable timer, and flow recorder. Minimally, a working motor/blower unit and flow recorder are required. Timing and on-off operation can be done manually if necessary. The motor/blower and the flow recorder should be checked as follows:

- The motor/blower unit should be removed and inspected. All wires should be properly connected. Motor brushes last for 400-500 hours of operation. They should be replaced when their viability is questionable or if the unit will be required to operate for over 400 hours. Be sure to seat the brushes properly when they are changed. Run the hi-vol at one-half voltage for about 20 minutes. A motor that is known or suspected to be broken should be discarded; it is essentially a disposable item. The thread fittings between the filter hood and motor/blower unit should be inspected for tightness, crossthreading, and leaks. Improper connections will cause inaccurate or variable flow readings. All black rubber faceplate gaskets on the filter holder faceplates should be inspected for wear and compressibility. A thin, hard, compressed gasket should be replaced because a good seal on the filter cannot be obtained with a gasket in this condition.

- The flow recorder hoses should be checked for deterioration. The recorder should be plugged in to make certain it works. The pen and arm assembly must be functional; spares can be obtained. All nuts, screws, and latches should be tightened. The unit should be plugged in and run for a while to detect the development of any problems.

- For other routine maintenance and specific preparations procedures, see the Sierra Instruments Operators Manual.

4.5.3 Sampler Installation

After determining the proper sampling location, drive four angle-iron legs into the ground; then attach the hi-vol unit with 5/16-inch bolts and level it. Hole drilling may be required. The power connections should be inside the housing. The hi-vol filter must be positioned at least 2 meters above ground.

4.5.4 Calibration

For a detailed discussion of calibration procedures for the particular hi-vol unit being used, refer to the Sierra Instruments Operators Manual, EPA Region VIII Policy (Max 1983), and EPA Quality Assurance Handbook, Section 2.2.2 (1985). Here is a synopsis of the calibration procedure used by the PIT.

- The variable resistance calibration unit should be installed on the sampler head by tightening the wingnuts.
- A slack tube manometer is connected to the orifice pressure tap and zeroed. Instructions for the use of the Dwyer slack tube manometer are in the calibration kit.
- The flow controller must be bypassed during calibration by disconnecting it and running power directly to the motor/blower unit. The flow recorder must also be plugged in.
- Turn the hi-vol sampler on and wait approximately 5 minutes for the brushes to seat and for temperature equilibrium to be achieved.
- Turn the resistance knob on the orifice unit until the desired manometer reading in inches H_2O is obtained for 60 cfm. The manometer readings on both sides should be added to obtain the reading. The calibration curve, provided by the EPA-ESD Quality Assurance Laboratory, is used to determine the manometer reading (inches of H_2O that corresponds to 60 cfm). Other calibration points typically used are 50, 30, and 20 cfm.
- Once the reading is exact, let the unit run for a few minutes to be sure the flow has stabilized.
- Zero the flow recorder. Gently blow into the pressure tube to be sure the pen rests on zero after descending across the chart. Install a clean chart and connect the tube to the pressure tap on the motor/blower unit. The pen should rise corresponding to the flow rate because the flow recorder is being calibrated, not the flow controller. The flow recorder is used as a relative indicator of constant flow over the sampling period and will usually not correspond to the correct calibration flow rate. After a brief stabilization period (3-5 minutes), check the manometer reading to be sure it is correct, and manipulate the flow recorder with a screwdriver in the center slot in a clockwise direction to draw a line on the chart corresponding to the

desired flow rate. Label with date, time, flow rate, EPA unit ID, and initials. This is the official calibration record. After each calibrated flow rate is obtained, disconnect the water manometer from the orifice pressure tap and attach it to the motor/blower unit tap. Obtain a reading and record it as a check for future reference.

- o Repeat the above procedure for all desired flow rates on the same flow recorder chart.
- o Turn off the hi-vol unit and remove the orifice calibration unit. Install a new calibration filter and flow recorder chart and reattach the orifice unit.
- o Reconnect the flow controller and all the other accessories on the hi-vol.
- o To calibrate the flow controller, turn on the hi-vol timer switch, let the unit stabilize, and adjust the orifice unit wide open.
- o Trim the calibration pot in the flow controller (see instruction manual) so that the exact reading is obtained on the flow recorder chart as the reading made on the calibration chart for 40 cfm. In conjunction with this, the water manometer can be hooked up to the meter unit pressure tap. As a check, the same reading should be obtained on the water manometer for 40 cfm as obtained during the preceding calibration procedures if the flow controller is calibrated properly.
- o The hi-vol should now be calibrated to 40 cfm, and it is ready for operation.

4.5.5 Sample Period

The sample period, project duration, and sampling days should be determined in advance in consultation with the EPA project officer (PO) and the ESD Air QA Lab personnel. Most hi-vol sampling is done for a continuous 24-hour period. The longest possible project duration will be beneficial to obtain a representative sample group. Variable weather conditions are an important factor to consider.

4.5.6 Filter Installation and Change

Filter numbers should be documented on the EPA data cards before installation on the hi-vol. Be sure to wear surgical gloves during all other sampling activities. Put a filter, number-side down, on the stainless steel screen of a filter cartridge assembly, assemble the cartridge, and snap on the cover. This assembly should be done in the FIT vehicle with the windows rolled up. Attach the filter cartridge to the hi-vol head by tightening the wingnuts. Take off the snap-on metal cover and close and padlock the shelter hood. When changing filters, use clean hands and fold the exposed filter lengthwise so that only the particulate surfaces are touching. Do not introduce any extraneous

contamination or particulate matter to the filter, and be careful not to loosen any sample. Immediately put the filter into an EPA Data Record folder, be sure all documentation is complete, and store in a dated EPA envelope.

The filter material typically used for collection of TSP is fiberglass, which is non-hygroscopic and, consequently, easy to desiccate to a constant weight prior to sampling. If the filters will be submitted for analysis of heavy metal concentration in the TSP, cellulose filters should be used. Cellulose does not contain heavy metals that will contaminate the field blank; however, it is difficult to dry the cellulose ester to constant weight. This problem can be overcome by placing the filters in a constant temperature and relative humidity environment for weighing.

4.5.7 Meteorological Station

The FIT has meteorological (MET) stations that are ideally suited and required for a hi-vol air-sampling project. Corresponding meteorological data must be known to obtain corrected flow calculations and to correlate environmental conditions to samples. The siting criteria, as discussed in the EPA Quality Assurance Handbook, Volume 4 (1985), should be referenced before setting the MET station on-site. Included with the MET equipment are complete operating instructions, which should be studied to obtain familiarity with the use of all the equipment before going into the field. All equipment must be calibrated per the instruction manuals. The charts generated by the meteorograph and the wind speed/direction records should be checked daily and changed if necessary (the meteorograph should be run with the 1-day gear in place). The time, the date, and the sampler's initials should be recorded on the charts daily.

4.6 QUALITY ASSURANCE

In order for air monitoring data to be useful, they must be of acceptable quality. The dissemination of poor quality data can lead to incorrect decisions with regard to environmental standards and regulatory actions. The essentials of complete QA program are described in detail in the EPA Quality Assurance Handbook (1985), Vol. 2, Sections 2.0 and 2.2.

4.6.1 Filter Preparation

For QA purposes, the ESD Air QA Lab will prepare any filters necessary for all FIT sampling projects. The filters will be equilibrated, marked, weighed, and packaged properly for convenient field use. After completing the sampling program, the exposed filters and flow rates for each sample will be submitted to the QA Lab, where the final weighing and TSP determinations will be conducted. All unused filters should also be returned to the lab.

4.6.2 Chain of Custody

Chain of custody (COC) procedures for ambient air samples are related in detail in the EPA Quality Assurance Handbook (1985), Vol. 2, Section 2.0.6, pp. 1-11. All FIT samplers must use these COC procedures and are urged to become familiar with this handbook section. All filters must be accounted for and archived by the FIT personnel.

4.6.3 Documentation

Rigorous maintenance of all documentation as the field program progresses is essential for legal purposes and for ensuring data reliability. The operator who starts the sampler is responsible for recording the following information:

- o Filter number.
- o Station designation, location, and address.
- o EPA Sampler ID number.
- o Starting time.
- o Chart installation and starting time.
- o Initial flow recorder and manometer measurements.
- o Summary of conditions that may affect sampling.

The operator who removes the sample is responsible for recording the following information:

- o Stop time and elapsed time (if available).
- o Final flow rate and volume (must be calculated).
- o Date and initials.
- o Remove all charts, date, and initial.
- o Summary of existing conditions.

Most of this information is recorded on the EPA hi-vol data record.

All calibrations should be recorded. An official log book should be kept daily for all project-related information.

4.6.4 Blank Samples

Normally, when the ESD Air QA Lab prepares the filters required for a FIT project, a sufficient number of extra filters will be weighed and included to be used as field blanks. These filters will be carried into the field daily in the filter folders designated for each sampling site. The filters are never removed from their folders. At the end of the

project, the number of blanks deemed appropriate (usually at least two) will be prepared from these extra filters and submitted with the actual samples.

4.6.5 Precision and Accuracy

An important requirement of the QA program for any FIT hi-vol TSP monitoring project is the assessment of precision and accuracy of reported monitoring data. These activities are described in the EPA Quality Assurance Handbook (1985), Vol. 2, Sections 2.0.8, 2.2.8, and 2.2.9. The assessment of precision requires collocating two hi-vol samplers, and the assessment of accuracy requires periodic flow audits. The FIT PO must be aware of these activities in preparing the sample plan, in evaluating siting, and in scheduling the field program.

4.7 CALCULATIONS

All calculations necessary to obtain the proper data required for a hi-vol program and TSP analysis are outlined in the EPA Region VIII Policy (May 1983). The reference must be used in Region VIII for all calculations. Follow the instructions pertaining to a flow-controlled hi-vol unit. In addition to the routine calculations to convert daily data to standard conditions, it is important that a calibration curve be drawn for the unit. An example of a calibration curve can be found in Appendix E. Final TSP calculations cannot be performed until gravimetric analysis data are received from the analyzing laboratory. See Table 4-4 for an example of a daily data sheet.

5 SOURCES OF ASSISTANCE

If additional assistance or clarification is needed for any aspect of a hi-vol air sampling program, contact E & E corporate headquarters in Buffalo, New York, or the ESD Air QA Lab, Denver Federal Center.

TABLE 4-4. Routine Calculations for a TSP Hi-Vol Sampling Program

SITE: _____ TDD: _____

DATE SAMPLE TAKEN _____

SITE DESIGNATION _____ _____ _____ _____

UNIT (EPA ID)	_____	_____
1. QR(m^3/min)	_____	_____
2. TA(°K)	_____	_____
3. PA(mmHg)	_____	_____
4. Sample Period (min)	_____	_____
5. QSTD(m^3/min)	_____	_____
6. VOL(STD m^3)	_____	_____

Calculations by _____ Date: _____

1. $QR = \frac{Ii + IF}{2}$ (Readings from calibration graph--or average reading from flow recorder daily chart.)
2. 273° + ambient °C, °F = (9/5 °C) + 32
3. Inches Hg--mmHg
4. Hours x 60
5. $QSTD = QR \left|\frac{PA\ TSTD}{TA\ PSTD}\right|^{1/2}$
6. V(STD/m^3) = QSTD (STD m^3/min) x T (min)

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APPENDIX A

40 CFR 58

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(OMITTED)

STANDARD OPERATING PROCEDURE FOR SOIL SAMPLING

REVISED: JANUARY 1993

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1 INTRODUCTION

This document describes the procedures for the collection of representative soil samples. Representative sampling ensures the accurate characterization of site conditions. Analysis of soil samples may determine pollutant concentrations and its risk to public health, welfare, or the environment; extent of contamination; and confirmation of remediation standards.

2 SCOPE

Included in this discussion are procedures for obtaining representative samples, quality assurance/quality control measures, proper documentation of sampling activities, and recommendations for personnel safety.

3 METHOD SUMMARY

Soil samples may be recovered using a variety of methods and equipment. These are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type.

Near-surface soils may be easily samples using a spade, trowel, or scoop. Sampling at greater depths may be performed using a hand auger, a power auger, or, if a test pit is required, a backhoe.

All sampling devices should be cleaned using pesticide grade acetone (assuming that acetone is not a target compound) or methanol, then wrapped in cleaned aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until it is needed. Each sampler should be used for one sample only. However, dedicated samples may be impractical if there are a large number of soil samples required. In this case, samplers should be cleaned in the field using the decontamination procedure outlined in E & E's SOP for Equipment Decontamination.

4 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The chemical preservation of solids is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time (see Table 3).

Soil samples should be handled according to the procedures outlined in E & E's SOP for Sample Packaging and Shipping.

5 POTENTIAL PROBLEMS

Potential problems with soil sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, the disturbance of the matrix resulting in compaction of the sample, and inadequate homogenization of the sample where required, resulting in variable, nonrepresentative results. Specific advantages and disadvantages of soil sampling equipment are given in Table 1.

6 SOIL SAMPLING EQUIPMENT

Soil Sampling Equipment List

- o Trier
- o Scoop
- o Trowel
- o Spatula
- o Tulip bulb planter
- o Spade or shovel
- o Vehimeyer soil sampler outfit
  - tubes
  - points
  - drive head
  - drop hammer
  - fuller jack and grip
- o Soil coring device
- o Ekman dredge
- o Ponar dredge
- o Thin-wall tube sampler
- o Split spoon sampler
- o Shelby tube sampler
- o Laskey sampler
- o Bucket auger
- o Hand-operated power auger
- o Continuous-flight auger
- o Dutch auger
- o Eijkelcamp stoney soil auger
- o Backhoe

Sampling Support Equipment and Documentation List

- o Sampling plan
- o Sample location map
- o Safety equipment, as specified in the Health and Safety Plan
- o Decontamination supplies and equipment, as described in the Work Plan
- o Compass

- o Tape measure
- o Survey stakes or flags
- o Camera
- o Stainless steel buckets or bowls
- o Sample containers, precleaned (i.e., I-Chem)
- o Logbook
- o Chain-of-Custody forms
- o Canvas or plastic sheet
- o Soil gas probes
- o Infiltrometer
- o Pounding sleeve
- o Extension rods
- o T-Handle

Labeling, Packaging, and Shipping Supplies

- o Coolers
- o Labels for sample containers and coolers (i.e., "9" labels, "UP" labels, etc.)
- o Ice
- o Plastic bags for sample containers and ice
- o ESC paint cans and clamps for PCB sampling
- o Vermiculite
- o Duct and strapping tape
- o Federal Express airbills and pouches

6.1 GEOPHYSICAL EQUIPMENT

Geophysical techniques can be integrated with field analytical and soil sampling equipment to help define areas of subsurface contamination. For a description of the geophysical techniques and associated applications refer to E & E's SOP for Surface Geophysical Techniques.

7 REAGENTS

This procedures does not require the use of reagents except for decontamination of equipment, as required. Refer E & E's SOP for Equipment Decontamination, and site-specific work plan for proper decontamination procedures and appropriate solvents.

8 PROCEDURES

8.1 OFFICE PREPARATION

1. The preparation of a Health and Safety Plan is required prior to any sampling. The plan must be approved and signed by the Corporate Health and Safety Officer or his/her designee (i.e.,

the Regional Safety Coordinator (RSC)).

2. Prepare a sampling plan to meet the data quality objectives (DQO) of the project in accordance with contract requirement. Review available background information (i.e., topographic maps, soil survey maps, geologic maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling method to be employed, and the type and amounts of equipment and supplies required.

3. Obtain necessary sampling and monitoring equipment (see Section 6), decontaminate or preclean the equipment, and ensure that it is in working order.

4. Contact delivery service to confirm ability to ship all equipment and samples. Determine if shipping restrictions exist.

5. Prepare schedules and coordinate with staff, clients, and regulatory agencies, if appropriate.

8.2 FIELD PREPARATION

1. Identify local suppliers of sampling expendables (e.g., ice, plastic bags) and overnight delivery services (e.g., Federal Express).

2. Decontaminate or preclean all equipment before soil sampling, as described in E & E's SOP for Equipment Decontamination, or as deemed necessary.

3. A general site survey should be performed prior to site entry in accordance with the Health and Safety Plan followed by a site safety meeting.

4. Identify and stake all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner or field team prior to soil sampling.

8.3 REPRESENTATIVE SAMPLE COLLECTION

The objective of representative sampling is to ensure that a sample or group of samples adequately reflect site conditions.

8.3.1 Sampling Approaches

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Each approach is defined below. Table 2 summarizes the following sampling approaches and ranks

them from most to least suitable based on the sampling objective.

8.3.1.1 Judgmental Sampling

Judgmental sampling is based on the subjective selection of sampling locations relative to historical site information, on-site investigation (site walk-over), etc. There is no randomization associated with this sampling approach because samples are primarily collected at areas of suspected highest contaminant concentrations. Therefore, any statistical calculations based on the sampling results would be unfairly biased.

8.3.1.2 Random Sampling

Random sampling involves the arbitrary collection of samples within a defined area. Refer to USEPA, 1984 and USEPA, February 1989 for a random number table and guidelines on selecting sample coordinates. The arbitrary selection of sample locations requires each sample location to be chosen independently, and that results in all locations within the area of concern have an equal chance of being selected. To facilitate statistical probabilities of contaminant concentration, the area of concern must be homogeneous with respect to the parameters being monitored. Thus, the higher the degree of heterogeneity, the less the random sampling approach will reflect site conditions. Refer to Figure 1 for the random sampling approach.

8.3.1.3 Stratified Random Sampling

Stratified random sampling primarily relies on historical information and prior analytical results to divide the area of concern into smaller sampling areas or "strata". Strata can be defined by several factors, such as: sampling depth, contaminant concentration levels, and contaminant source areas. Sampling locations should be selected within a strata using random selection procedures. Figure 2 illustrates a stratified random sampling approach.

8.3.1.4 Systematic Grid Sampling

Systematic grid sampling involves dividing the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersection of the grid lines or "nodes". The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the area of concern and the number of samples to be collected. Refer to Figure 3 for the systematic grid sampling approach.

8.3.1.5 Systematic Random Sampling

Systematic random sampling involves dividing the area of concern into smaller sampling areas as described in Section 8.3.1.4. Samples are collected within each grid cell using random selection procedures.

Figure 4 illustrates a systematic random sampling approach.

8.3.1.6 Search Sampling

Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed clean-up standards or "hot spots". The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots. Figure 5 illustrates a search sampling approach.

8.3.1.7 Transect Sampling

Transect sampling involves establishing one or more transect lines, parallel or non-parallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is the relative ease of establishing and relocating transect lines versus an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected. Refer to Figure 6 for the transect sampling approach.

8.3 REPRESENTATIVE SAMPLE COLLECTION

8.3.2 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, and scoops. The surface material can be removed to the required depth with this equipment; then stainless steel or plastic scoops can be used to collect the sample.

This method can be used in most soil types but is limited to sampling near surface areas. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sampling technician. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required (i.e., for VOAs). A stainless steel scoop, lab spoon, or plastic spoon will suffice in most other applications. Care should be exercised to avoid the use of devices plated with chrome or other materials. Plating is particularly common with garden implements such as potting trowels.

The following procedure is used to collect the soil samples:

1. Carefully remove the top layer of soil to the desired sample depth with a precleaned spade.

2. Using a precleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the shovel.

3. Transfer sample into an appropriate sample container with a stainless steel or plastic lab spoon, or equivalent. If composite samples are to be collected, place the soil sample in a stainless steel or plastic bucket, and mix thoroughly to obtain a homogeneous sample representative of the entire sampling interval. Then, place soil sample into labeled containers. Caution: Never composite VOA samples.

4. Samples for volatile organic analysis will be collected directly from the bottom of the hole before mixing the sample, to minimize volatilization of contaminates.

5. Check that the VOA vial Teflon liner is present in the cap, if required. Fill the VOA vial fully to the top to reduce headspace. Secure the cap tightly. The chemical preservation of solids is generally not recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time.

6. Check to be sure that enough sample has been collected for the desired analysis, as specified in the Sampling Plan.

7. Decontaminate equipment between samples according to E & E's SOP for Equipment Decontamination.

8. Fill in the hole and replace grass turf if necessary.

9. Collect QA/QC samples as specified, according to the work plan.

8.3.3 Sampling at Depth with Augers and Thin-wall Tube Samplers

This system consist of an auger, a series of extensions, a T-handle, and a thin-walled tube. The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The auger tip is then replaced with a tube core sampler, lowered down the borehole, and driven into the soil at the completion depth. The core is then withdrawn and the sample collected.

Several augers are available (Appendix A). These include: bucket type, continuous flight (screw), and porthole augers. Bucket types are better for direct sample recovery as they provide a large volume of sample in a short time. When continuous-flight augers are used, the sample can be collected directly off the flights, usually at 5-foot intervals. The continous-flight augers are satisfactory for use when a composite of the complete soil column is desired. Posthole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil.

The following procedure will be used for collecting soil samples with the hand auger:

1. Attach the auger bit to a drill rod extension, and attach the T-handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first 3 to 6 inches of surface soil for an area approximately 6 inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a canvas or plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect sample after the auger is removed from boring and proceed to Step 10.

5. Remove auger tip from dill rods and replace with a precleaned thin-wall tube sampler. Install proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring, as the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler, and unscrew the drill rods.

8. Remove the cutting tip and core from the device.

9. Discard the top of the core (approximately 1 inch), as this represents material collected before penetration of the layer in question. Place the remaining core into the sample container.

10. If required, ensure that a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Place the sample bottle in a plastic bag, and put on ice to keep the sample at 4°C.

11. Carefully and clearly label the container with the appropriate sample tag addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping.

12. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Verify that the chain-of-custody form is correctly and completely filled out.

13. Record the time and date of sample collection, as well as a description of the sample in the field logbook.

14. If another sample is to be collected in the sample hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

15. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

16. Decontaminate the sampling equipment as per E & E's SOP for Equipment Decontamination.

8.3.4 Sampling at Depth with a Trier

1. Insert the trier (Appendix B) into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample. Extraction of samples might require tilting of the containers.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. Transfer the sample into a suitable container with the aid of a spatula and brush.

5. If required, ensure that a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping.

6. Carefully and clearly label the container with the appropriate sample tag addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping.

7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.

9. Abandon the hole according to applicable regulations.

Generally, shallow holes can simply be backfilled with the removed soil material.

10. Decontaminate sampling equipment as per E & E's SOP for Equipment Decontamination.

8.3.5 Sampling at Depth with a Split Spoon (Barrel) Sampler

The procedure for split spoon sampling describes the extraction of undisturbed soil cores of 18 or 24 inches in length (Appendix C). A series of consecutive cores may be sampled to give a complete soil column or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom the the augured hole and the core extraction.

This sampling device may be used to collect such information as soil density. All work should be performed in accordance with ASTM D 1586-84, Penetration Test and Split Barrel Sampling of Soils.

1. Assemble the sampler by aligning both sides of the barrel and then screwing the bit on the bottom and the heavier head piece on top. Install a retaining cap in the head piece if necessary.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a sledge hammer or well ring, if available, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.

4. Record the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.

5. Withdraw the split spoon and open by unscrewing the bit and head. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is available in 2- and 3 1/2-inch diameters. The required sample volume may dictate the use of the larger barrel. If needed, stainless steel or Teflon sleeves can be used inside the split spoon. If sleeves removed from the split spoon are capped immediately, volatization of contaminants can be reduced. When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974).

6. Cap the sample container, place in a double plastic bag and attach the label and custody seal. Record all pertinent data in the field logbook and complete the sample analysis request form and chain-of-custody record before taking the next sample.

7. If required, preserve or place the sample on ice.

8. Follow proper decontamination procedures and then deliver samples to the laboratory for analysis.

8.3.6 Test Pit/Trench Excavation

These relatively large excavations are used to remove sections of soils, when detailed examination of soil characteristics (horizontal, structure, color, etc.) are required. It is the least cost-effective sampling method due to the relatively high cost of backhoe operation.

1. Prior to any excavations with a backhoe, it is important to ensure that all sampling locations are clear of utility lines and poles (subsurface as well as above surface).

2. Using the backhoe, a trench is dug to approximately 3 feet in width and approximately 1 foot below the cleared sampling depth. Place removed or excavated soils on canvas or plastic sheets, if necessary. Trenches greater than 4 feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.

3. A shovel is used to remove a 1- to 2-inch layer of soil from the vertical face of the pit where sampling is to be done.

4. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose soil for sampling. Samples are removed and placed in an appropriate container.

5. If required, ensure that a Teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping.

6. Carefully and clearly, label the container with the appropriate sample tag addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping.

7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.

9. Abandon the hole according to applicable State regulations. Generally, excavated holes can simply be backfilled with the removed soil material.

10. Decontaminate sampling equipment including the backhoe bucket, as per E & E's SOP for Equipment Decontamination.

8.4 SAMPLE PREPARATION

In addition to sampling equipment, representative sample collection includes sample quantity, volume, preservation, and holding time. Sample preparation refers to all aspects of sample handling after collection. How a sample is prepared can affect its representativeness. For example, homogenizing can result in a loss of volatiles and is therefore inappropriate when volatile contaminants are the concern. Table 3 provides guidelines on appropriate sample containers, minimum volume requirements, preservatives, and holding times.

8.4.1 Sample Quantity and Volume

The volume and number of samples necessary for site characterization will vary according to the budget, project schedule, and sampling approach.

8.4.2 Sample Preservation and Holding Time

Sample preservation and holding times are as discussed in section 4.

8.4.3 Removing Extraneous Material

Discard materials in a sample which are not relevant for site or sample characterization (i.e., glass, rocks, or leaves), since their presence may introduce an error in analytical procedures.

8.4.4 Homogenizing Samples

Homogenizing is the mixing of a sample to provide a uniform distribution of the contaminants. Proper homogenization ensures that the containerized samples are representative of the total soil sample collected. All samples to be composited or split should be homogenized after all aliquots have been combined (Figure 7). Do not homogenize samples for volatile compound analysis.

8.4.5 Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provides an average concentration of contaminants over a certain number of sampling points. Compositing dilutes high concentration aliquots, therefore detection limits should be reduced accordingly. If the composite area is heterogeneous in concentration and its composite value is to be compared to a particular action-level, then that action-level must be divided by the total number of aliquots

making up the composite for accurate determination of the detection limit.

8.4.6 Splitting Samples

Splitting samples (after preparation) is performed when multiple portions of the same samples are required to be analyzed separately. Fill the sample containers simultaneously with alternate spoonfuls of the homogenized sample.

8.5 POST OPERATIONS

8.5.1 Field

1. Decontaminate all equipment according to E & E's SOP for *Equipment Decontamination*.

8.5.2 Office

1. Organize field notes into a report format and transfer logging information to appropriate forms.

9 CALCULATIONS

There are no specific calculations required for these procedures.

10 QUALITY ASSURANCE/QUALITY CONTROL

The objective of QA/QC is to identify and implement methodologies which limit the introduction of error into sampling and analytical procedures.

10.1 SAMPLING DOCUMENTATION

10.1.1 Soil Sample Label

All soil samples shall be documented in accordance with E & E's SOP for Sample Packaging and Shipping. The soil sample label is filled out prior to collecting the sample, and should contain the following:

1. Site name or identification.
2. Sample location and identifier.
3. Date samples were collected in a day, month, year format (e.g., 03 Jan 88 for January 3, 1988).
4. Time of sample collection, using 24-hour clock in the hours: minutes format.
5. Sample depth interval. Units used for depths should be in feet and 10ths of feet.
6. Preservatives used, if any.

7. Analysis required.
8. Sampling personnel.
9. Comments and other relevant observations (e.g., color, od , sample technique).

10.1.2 Logbook

A bound field notebook will be maintained by field personnel to record daily activities, including sample collection and tracking information. A separate entry will be made for each sample collected. These entries should include information from the sample label and a complete physical description of the soil sample including texture, color (including notation of soil mottling), consistency, moisture content, cementation, and structure.

10.1.3 Chain-of-Custody

Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Refer to E & E's SOP for Sample Packaging and Shipping for directions on filling out this form.

10.2 SAMPLING DESIGN

1. Sampling situations vary widely and therefore no universal sampling procedure can be recommended. However, a sampling plan should be implemented before any sampling operation is attempted with attention to contaminant type and potential concentration variations.

2. Any of the sampling methods described here should allow a representative soil sample to be obtained if the sampling plan is properly designed.

3. Consideration must also be given to the collection of a sample representative of all horizons present in the soil. Selection of the proper sampler will facilitate this procedure.

4. A stringent QA project plan should be outlined before any sampling operation is attempted. This should include, but not be limited to, properly cleaned samplers and sample containers, appropriate sample collection procedures, chain-of-custody procedures, and QA/QC samples.

11 DATA VALIDATION

The data generated will be reviewed according to QA/QC considerations identified in Section 10.

11.1 QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

QA/QC samples are used to identify error due to sampling and/or

analytical methodologies and chain-of-custody procedures.

11.1.1 Field Duplicates (Replicates)

Field duplicates are collected from one location and treated as separate samples throughout the sample handling and analytical processes. These samples are used to assess total error for critical samples with contaminant concentrations near the action level.

11.1.2 Collocated Samples

Collocated samples are generally collected 1.5 to 3.0 feet away from selected field samples to determine both local soil and contaminant variations on-site. These samples are used to evaluate site variation within the immediate vicinity of sample collection.

11.1.3 Background Samples

Background or "clean" samples are collected upgradient from the contamination area. These samples provide a standard for comparison of on-site contaminant concentration levels.

11.1.4 Rinsate (Equipment) Blanks

Rinsate blanks are collected by pouring analyte-free water (i.e., laboratory de-ionized water) on decontaminated sampling equipment to test for residual contamination. These samples are used to assess potential cross-contamination due to improper decontamination procedures.

11.1.5 Performance Evaluation (PE) Samples

Performance evaluation samples are generally prepared by a third party, using a quantity of analyte(s) known to the preparer but unknown to the laboratory. The percentage of analyte(s) identified in the sample is used to evaluate laboratory procedural error.

11.1.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

MS/MSD samples are spiked in the laboratory with a known quantity of analyte(s) to confirm percent recoveries. They are primarily used to check sample matrix interferences.

11.1.7 Field Blanks

Field blanks are prepared in the field with certified clean sand, soil, or water. These samples are used to evaluate contamination error associated with sampling methodology and laboratory procedures.

11.1.8 Trip Blanks

Trip blanks are prepared prior to going into the field using certified clean sand, soil, or water. These samples are used to assess error associated with sampling methodology and analytical procedures for volatile organics.

12 HEALTH AND SAFETY

12.1 HAZARDS ASSOCIATED WITH ON-SITE CONTAMINANTS

Depending on site-specific contaminants, various protective programs must be implemented prior to soil sampling. The site Health and Safety Plan should be reviewed with specific emphasis placed on a protection program planned for direct contact tasks. Standard safe operating practices should be followed, including minimization of contact with potential contaminants in both the vapor phase and solid matrix by using both respirators and disposable clothing.

Use appropriate safe work practices for the type of contaminant expected (or determined from previous sampling efforts):

Particulate or Metals Contaminants

- Avoid skin contact with, and ingestion of, soils and dusts.
- Use protective gloves.

Volatile Organic Contaminants

- Presurvey the site with an HNU 101 or OVA 128 prior to taking soil samples.
- If monitoring results indicate organic constituents, sampling activities may be conducted in Level C protection. At a minimum, skin protection will be afforded by disposable protective clothing.

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TABLES

SOIL SAMPLING EQUIPMENT
REPRESENTATIVE SAMPLING APPROACH COMPARISON
STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS,
AND VOLUME REQUIREMENTS

TABLE 1: SOIL SAMPLING EQUIPMENT

Equipment	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop, trowel, or spatula	Soft surface soil	Inexpensive; easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0-6 in.	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; not useful for hard soils.
Spade or shovel	Medium soil, 0-12 in.	Easy to use and decontaminate; inexpensive; can result in sample mixing and loss of VOC.
Vehimeyer soil outfit	Soil, 0-10 ft	Difficult to drive into dense or hard material; may be difficult to pull from ground.
Soil coring device	Soft soil, 0-24 in.	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-wall tube sampler	Soft soil, 0-10 ft	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used to help maintain integrity of VOA samples; easy to decontaminate; can be difficult to remove cores from sampler.

Table 1 (Cont.)
Page 2

Equipment	Applicability	Advantages and Disadvantages
Split spoon sampler	Soil, 0 in.-bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.
Shelby tube sampler	Soft soil, 0 in.-bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Laskey sampler	Soil, 0 in.-bedrock	Excellent depth range; preserves soil cores; used in conjunction with drill rig for obtaining deep core; can be difficult to decontaminate.
Bucket auger	Soft soil, 3 in. - 10 ft	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 in.-15 ft	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires 2 or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).

Table 1 (Cont.)
Page 3

Equipment	Applicability	Advantages and Disadvantages
Continuous-flight auger	Soil, 0 in.-bedrock	Excellent depth range; easy to decontaminate; can be used on all soil samples; results in soil mixing and loss of VOC.
Dutch auger	Designed specifically for wet, fibrous, or rooted soils (marshes)	
Eijkelcamp stoney soil auger	Stoney soils and asphalt	
Backhoe	Soil, 0 in. - 10 ft.	Good depth range; provides visual indications as to depth of contaminants; allows for recovery of samples at specific depths; can result in loss of VOC and soil mixing; shoring required at depth.

kvk/[ENV]#4077

Note: 1) Samplers may not be suitable for soils with coarse fragments.

2) Augers are suitable for soils with limited coarse fragments; only the stoney auger will work well in very gravelly soil.

TABLE 2: REPRESENTATIVE SAMPLING APPROACH COMPARISON

Sampling Objective	Judgmental	Random	Stratified Random	Systematic Grid	Systematic Random	Search	Transect
Establish threat	1	4	3	2[a]	3	3	2
Identify Sources	1	4	2	2[a]	3	2	3
Delineate Extent of Contamination	4	3	3	1[b]	1	1	1
Evaluate Treatment and Disposal Options	3	3	1	2	2	4	2
Confirm Cleanup	4	1[c]	3	1[b]	1	1	1[d]

kvk/[ENV]#4078

1 - Preferred approach

2 - Acceptable approach

3 - Moderately acceptable approach

4 - Least acceptable approach

a - Should be used with field analytical screening

b - Preferred only where known trends are present

c - Allows for statistical support of cleanup verification if sampling over entire site

d - May be effective with compositing technique if site is presumed to be clean

Table 3

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
SW-846								
VOA[c]	14 days from date sampled	14 days from date sampled	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4°C (ice in cooler)	Add HCl until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[c]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,c]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,c]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 until pH <2 and cool to 4°C (ice in cooler)
Cyanide[c]	14 days from date sampled	14 days from date sampled	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH until pH >12 and cool to 4°C (ice in cooler)
Hexavalent chromium[a]	24 hours from time sampled	24 hours from time sampled	10 g	50 ml	8 oz. glass jar with Teflon-lined cap	125 ml polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)

Key at end of table.

Table 3

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
Total Organic Carbon (TOC)[a]	NA	28 days from date sampled	5 g	10 ml	8 oz. glass jar with Teflon-lined cap	125 ml polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
Total Organic Halides (TOX)[a,c]	NA	7 days from date sampled	100 g	200 ml	8 oz. glass jar with Teflon-lined cap	1 L amber glass bottle	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
Total Recoverable Petroleum Hydrocarbons[c]	28 days from date sampled	28 days from date sampled	50 g	1 L	8 oz. glass jar with Teflon-lined cap	1 L amber glass bottle	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
USEPA-CLP								
VOA[c]	10 days from date received	10 days from date received	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4°C (ice in cooler)	Add HCl until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[c]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,c]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,c]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 to pH <2 and cool to 4°C (ice in cooler)

Key at end of table.

Table 3

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
Cyanide[c]	12 days from date received	12 days from date received	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH to pH >12 and cool to 4°C (ice in cooler)
NYSDEC-CLP								
VOA[b,c]	7 days from date received	10 days from date received	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4°C (ice in cooler)	Add HCl until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[c]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 to pH <2 and cool to 4°C (ice in cooler)
Cyanide[c]	12 days from date received	12 days from date received	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH to pH >12 and cool to 4°C (ice in cooler)

Key at end of table.

Table 3

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
EPA Water and Waste								
Total dissolved solids (TDS)	NA	7 days from date sampled	NA	200 ml	NA	1 L polyethylene bottle with polyethylene-lined cap	NA	Cool to 4°C (ice in cooler)

Note: All sample bottles will be prepared in accordance with EPA bottle-washing procedures. These procedures are incorporated in E & E's Laboratory and Field Personnel Chain-of-Custody Documentation and Quality Assurance/Quality Control Procedures Manual, July 1987.

[a] Technical requirements for sample holding times have been established for water matrices only. However, they are also suggested for use as guidelines in evaluating soil data.

[b] Holding time for GC/MS analysis is 7 days if samples are not preserved.

[c] Maximum holding time for mercury is 28 days from time sampled.

[d] If one container has already been collected for PCBs analysis, then only one additional container need be collected for extractable organics, BNAs, or pesticides/PCBs analysis.

[e] Extra containers required for MS/MSD.

Key:

NA = Not applicable.

FIGURES

Figure 1: Random Sampling **

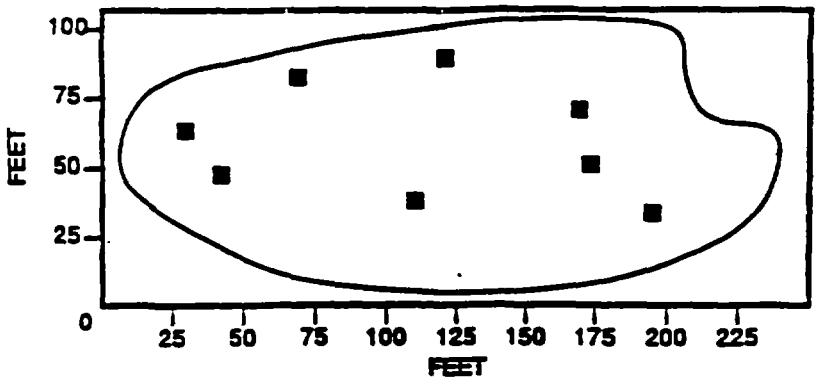


Figure 2: Stratified Random Sampling

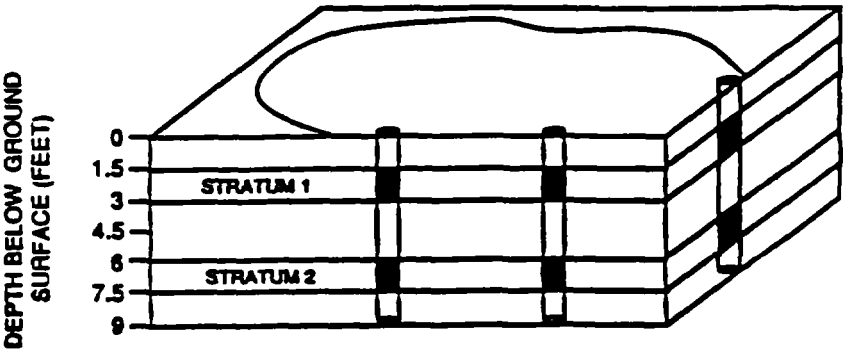
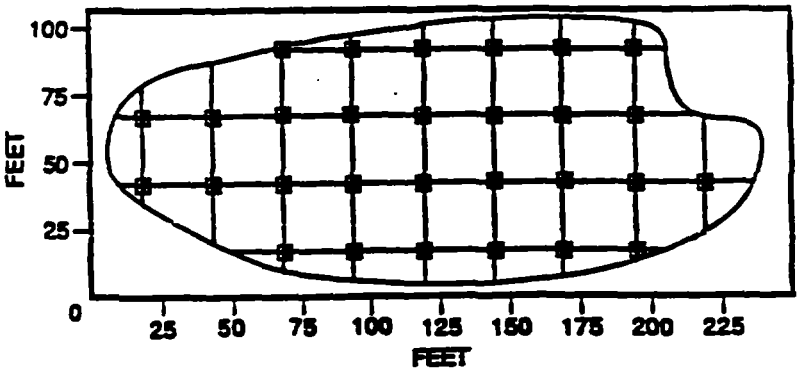


Figure 3: Systematic Grid Sampling **



** After U.S. EPA, February, 1989

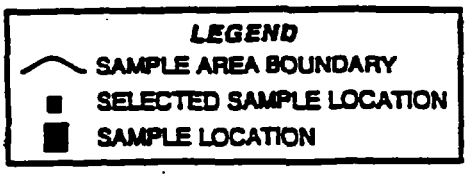


Figure 4: Systematic Random Sampling

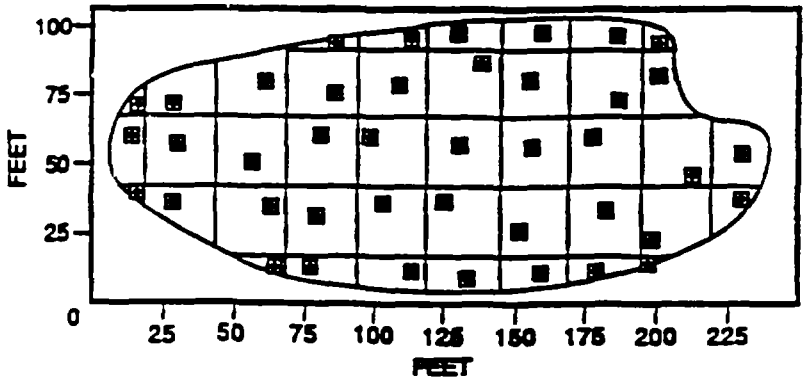


Figure 5: Search Sampling

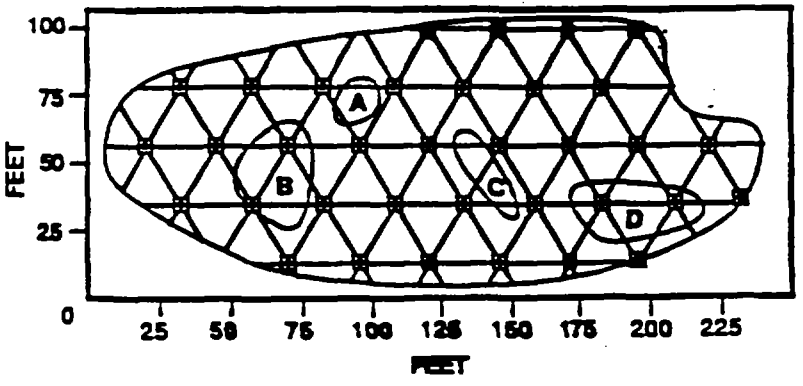
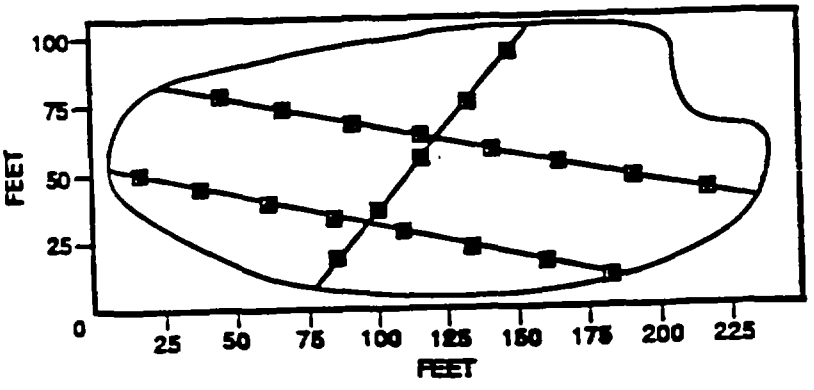
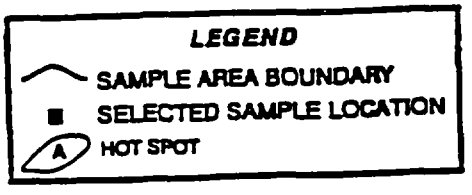
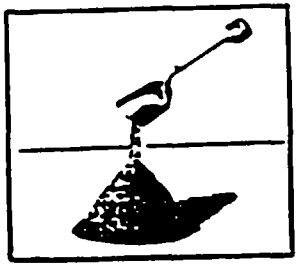


Figure 6: Transect Sampling



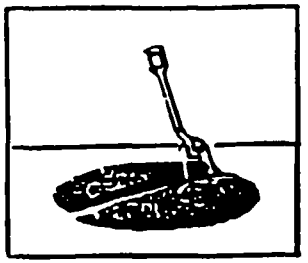
After: U.S. EPA, February, 1989





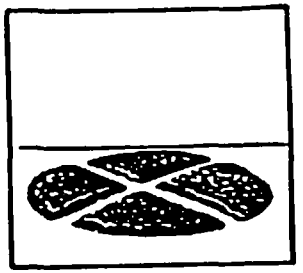
Step 1:

- **Cone Sample on Hard Clean Surface**
- **Mix by Forming New Cone**



Step 2:

- **Quarter After Flattening Cone**



Step 3:

- **Divide Sample into Quarters**

Step 4:

- **Remix Opposite Quarters**
- **Reform Cone**
- **Repeat a Minimum of 5 Times**

After: ASTM Standard C702-87

Figure 7: Quartering to Homogenize and Split Samples

13 REFERENCES

ASTM D 1586-67 (reapproved 1974), ASTM Committee on Standards, Philadelphia, PA.

ASTM D 1586-84. Penetration Test and Split Barrel Sampling of Soils.

Barth, D. S. and B. J. Mason, 1984, Soil Sampling quality Assurance User's Guide, EPA-600/4-84-043.

de Vera, E. R., B. P. Simmons, R. D. Stephen, and D. L. Storm, 1980, Samplers and Sampling Procedures for Hazardous Waste Streams, EPA-600/2-80-018.

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- o "Equipment Decontamination"
- o "Sample Packaging and Shipping"

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U.S. Environmental Protection Agency, 1984, Characterization of Hazardous Waste Sites - A Methods manual: Volume I, Site Investigations, Section 7: Environmental Monitoring Systems Laboratory, Las Vegas, Nevada, EPA/600/4-84/075.

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APPENDIX A

SAMPLING AUGERS

(a)

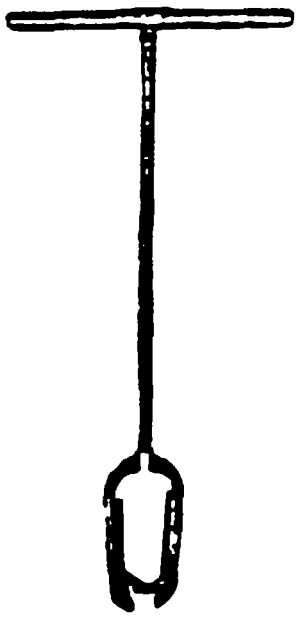
Ship Auger

(b)

Closed-Spiral Auger

(c)

Open-Spiral Auger

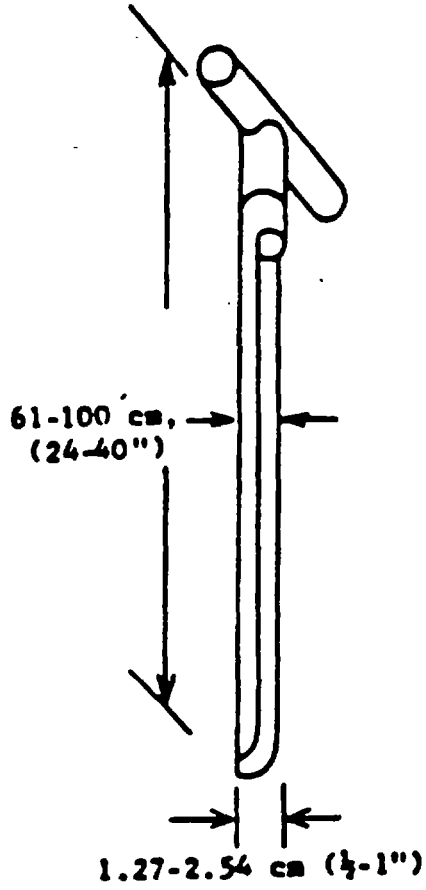


(d)

Iwan Auger

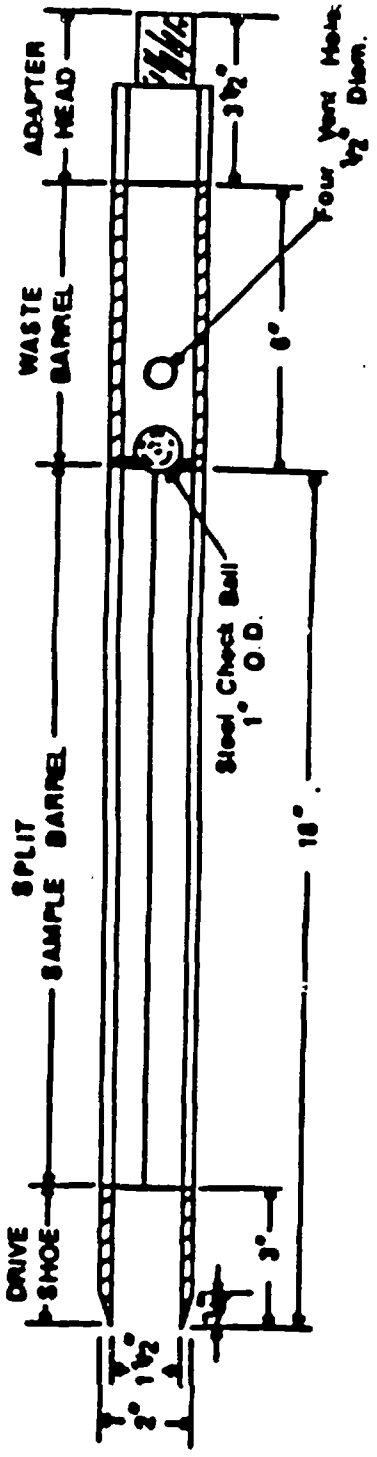
APPENDIX B

SAMPLING TRIER


61-100 cm,
(24-40")
1.27-2.54 cm (½-1")

APPENDIX C

SPLIT SPOON SAMPLER


ADAPTER HEAD
WASTE BARREL
SPLIT SAMPLE BARREL
DRIVE SHOE
Four Vent Holes ½" Diam.
Steel Check Ball 1" O.D.
3½"
6"
18"
3"

PB92-963405

OSWER Directive 9360.4-04
May 1992

COMPENDIUM OF ERT FIELD ANALYTICAL PROCEDURES

Sentex Scentograph Gas Chromatograph

Portable XRF Analyzer

Photoionization Detector -- HNU

Photovac 10A10 Portable Gas Chromatograph Operation

Photovac 10S50, 10S55, and 10S70 Gas Chromatograph Operation

Photovac GC Analysis for Air, Soil Gas, Water, and Soil

Micromonitor M200

Interim Final

Environmental Response Team
Emergency Response Division

Office of Emergency and Remedial Response
U.S. Environmental Protection Agency
Washington, DC 20460

Notice

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Each Standard Operating Procedure in this compendium contains a discussion on quality assurance/quality control (QA/QC). For more information on QA/QC objectives and requirements, refer to the *Quality Assurance/Quality Control Guidance for Removal Activities*, OSWER directive 9360.4-01, EPA/540/G-90/004.

Questions, comments, and recommendations are welcomed regarding the Compendium of ERT Field Analytical Procedures. Send remarks to:

Mr. William A. Coakley
Removal Program QA Coordinator
U.S. EPA - ERT
Raritan Depot - Building 18, MS-101
2890 Woodbridge Avenue
Edison, NJ 08837-3679

For additional copies of the Compendium of ERT Field Analytical Procedures, please contact:

National Technical Information Service (NTIS)
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161
(703) 487-4600

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List of Exhibits

Acknowledgments

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1.0 SENTEX SCENTOGRAPH GAS CHROMATOGRAPH: SOP #1702

1.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) primarily deals with the assessment of gaseous matrix samples. The Sentex Scentograph Gas Chromatograph (GC) can work in two detector configurations: the Electron Capture Detector (ECD) or the Argon Ionization Detector (AID). The ECD analyzes volatile chlorinated compounds, as it is very sensitive to electrophilic compounds such as chlorinated organics. The AID is a more universal detector, responding to most compounds with ionization potentials at or below 11.7 eV. It will respond to most aromatic compounds and many chlorinated compounds of environmental interest.

At present, only vapor-phase samples (e.g., soil gas samples, Tedlar bag gas samples, and ambient air samples) are analyzed through the activation of the instrument's internal sampling pump. The Sentex GC unit does have a syringe injection port, but this is not being used for any ERT applications at present. An optional purge and trap unit is available to determine purgeable organics in soil or water matrices. However, this SOP does not cover that capability.

1.2 METHOD SUMMARY

The initial step in Sentex Scentograph Gas Chromatograph sampling is to turn on and boot the Toshiba T1100 computer. The computer runs the data acquisition program and stores all parameters and data. Begin by inserting the program disk "A" and the data "B" disk into the appropriate upper and lower disk drives of the computer. Next, turn on the Sentex GC and, following the menu prompts, enter the GC operational parameters into the computer. Calibration analysis is then performed and stored. Calibration standards can be run either in the field, or in the laboratory prior to field sampling. In the latter case, a field calibration run must still be conducted to ensure that the lab calibrations are valid. A Tedlar bag of a standards mixture can be attached to the Sentex GC upper inlet port at this point, or the internal calibration gas cylinder can be used. Once calibration analysis is validated and stored, a Tedlar bag containing an unknown sample is attached to the Sentex GC's lower sampling inlet port, and the bag valve is opened. By selecting *Function #3* from the computer menu, a manual analysis can be run.

Once the sampling pump stops, the valve of the sample bag is closed, and the entire bag is removed from the inlet port and stored for future laboratory analysis.

While this procedure is standard for all Sentex GC sampling, actual operating conditions (e.g., detector used, column packing material, oven temperature) will vary as required by the sample matrix encountered, and by the physical and chemical nature of the samples analyzed. New operating parameters are determined as new target compounds are selected for analysis.

1.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The vast majority of Sentex applications are for soil gas analysis. These soil gas samples are collected and stored as outlined in ERT SOP #2149, Soil Gas Sampling.

1.4 INTERFERENCES AND POTENTIAL PROBLEMS

Since the Sentex units use gas chromatography, target compounds are identified by retention time indices (RTI). If the RTI of the sample peak(s) matches the RTI of the standard peak(s), it is assumed to be identical. If any non-target compound has the same RTI, it can be misidentified as a target compound. This problem occurs more frequently with the AID, since it will respond to any compound at or below 11.7 eV. Often, soil gas samples will have very high (ppm) levels of C_1 to C_6 hydrocarbons, as well as low (ppb) levels of target compounds. The AID will respond to these hydrocarbons, whose signal often can "swamp" or obscure the signal of the lower level target compounds. In this case, it is better to use the ECD. If this is not possible, a different GC column (one able to separate the target compounds from

the interference of the sample matrix) must be utilized. Moisture within the Tedlar bags will yield interference peaks that can obscure the resolution of the target compounds. It has not been determined whether the moisture itself, or contaminants in the moisture, yields these contamination peaks, but the effect is seen on both the AID and the ECD. Typically, when water is found in the Tedlar bags, the results of that bag's analysis are considered questionable. Typical ambient air relative humidity has no appreciable effect on the signal response.

1.5 EQUIPMENT/APPARATUS

A Sentex model Scentograph GC can be configured for AID or ECD. Interfaced with the unit is a Toshiba model T1100 lap top computer which runs the data acquisition program and stores all parameters and data. The Sentex unit has an internal battery pack which is charged from a Power Sonic Corporation model PSC-12400 (115 VAC to 12 VDC) charger. Attached to the T1100 is a Hewlett-Packard Model 2225P Think Jet printer which produces hard copies of chromatograms and peak data information. No other equipment is required to operate the Sentex Scentograph unit.

1.6 REAGENTS

The Sentex AID requires ultrahigh purity (99.99% or above) Argon as carrier gas. The ECD can use either ultrahigh purity Argon or Helium (these can be ordered from Scott Gas, Matheson or any other reliable vendor). Gas standards are purchased as certified mixtures from Scott Gas or Matheson, at fairly high concentrations (i.e., 1-50 ppm). These concentrations subsequently dilute to various concentrations that enable construction of a standard calibration curve. If the internal calibration cylinder is used, a low-level standard, such as 0.5-1.0 ppm, should be used.

If liquid phase standards are required, they must also be of the highest purity, such as Aldrich Gold Label or Supelco Environmental standards kits. If air is to be used for sample/standards dilutions, it must also be ultrahigh purity gas.

1.7 PROCEDURES

All operational parameters are entered from the T1100 computer and accessed from the operations menu, *Function #1*, which appears when the Sentex unit is turned on. Once the parameters are entered, run for calibration, *Function #4*. This is not a true calibration, since the Sentex calculates a pseudo-concentration against only one concentration. The calibration function is used only when operating parameters under *Function #1* are changed. In all other cases, it is ignored. After the first "junk" or noise peak is identified in the calibration run, the run is typically aborted. Occasionally, a Tedlar bag or the internal calibration gas cylinder can be sampled in the calibration mode, but this would be only for peak identification and semi-quantification purposes, since it is a single point calibration.

1.7.1 Calibration

The generation of calibration standards to be run in the field can be performed either in the field or in the laboratory prior to entering the field. If the latter is done, field standards must still be run to ensure calibration runs stored on the data disks are valid and close to standards run in the lab. Dilutions are typically made from the certified gas standards' cylinders using Hamilton 500, 1000 and 1500 cm^3 model "Super syringes", and from Tedlar sampling bags. Simple volumetric dilutions are made and the set of standards analyzed as if they were typical samples.

At least three concentrations of each standard must be run; however, more standards are run to establish the minimum ranges for the linear response of the Sentex detectors for each individual target compound. In the laboratory, a Multi-Channel Mass Flow Control can be used to meter selected flow rates from two to four separate compressed gas cylinders. Establish a continuous flow of a selected concentration of mixtures to either fill Tedlar bags for analysis or to create a flow-through-cell from which the Sentex GC can sample. This method has been used extensively to establish minimum and maximum detection levels in an efficient and timely manner.

1.7.2 Sampling

Follow this procedure for analyzing Tedlar bags with the Scentograph.

1. Insert Disks "A" and "B" into upper and lower T1100 disk drives respectively, and turn on Sentex GC.

2. Follow menu prompts and input GC parameters according to Table 1, Scentograph Operating Parameter Menu.

3. Select *Function #4* to run and store calibration analysis.

4. Attach Tedlar bag with unknown sample to lower sample inlet port and slide bag valve down to open.

5. Select *Function #3* to run a manual analysis. Type the sample name; press <ENTER> a second time to inject.

6. Immediately after the sampling pump stops, pull bag valve out to close; remove bag from inlet port.

7. To abort sample and calibration analysis, hold down the *Reset* key on the GC panel until the "Return" prompt appears.

8. Any changes in operating parameters entered in Function #1 must be followed by a calibration run prior to an analysis run.

1.8 CALCULATIONS

A calibration curve of at least three concentrations must be constructed for each target compound. A straight line equation in the form of $y = (m)(x) + b$ (where: x = concentration, y = area counts, m = slope and b = the intercept) is fit to the standards raw data. The (y), or the unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative of erroneous detector response. Alternatively, sample concentration can be calculated as shown below:

$$\text{Sample Conc.} = \frac{(\text{standard conc.})(\text{sample area})}{(\text{standard area})}$$

The Sentex performs a one point calibration for those compounds entered in the library. If the samples and library standard are in the linear range, this one point calibration is considered valid for screening purposes.

1.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following QA/QC protocols are applicable:

- Run a complete calibration curve daily.
- Duplicates of a standard, in the mid-range of the calibration curve and preferably close to sample results, should be run every 10 samples or so, to ensure detector response is constant.
- Run two to three duplicates for each sample and standard. In terms of area count and retention time values, these duplicate responses should be within 10-20% of each other.
- Matrix spikes, or spiking samples with known levels of standards, are not typically required, as the same Tedlar bag may be analyzed by other field instrumentation (e.g., Photovac, OVA, etc.) and/or collected onto traps for GC/MS confirmation. If Tedlar bags are used to prepare standards, the time of preparation should be noted.
- During sample analysis, one of the standards should be periodically re-analyzed to test for any sample loss in the bag over time.
- A performance evaluation (PE) sample is typically sent along with the samples to test for any loss or contamination from transit or handling during sampling.
- Send a trip blank of zero air along for analysis at the end of the sampling run to determine if any contamination of the Tedlar bags occurred during transit.

Table 1: Scentograph Operating Parameter Menu

#	Operating Parameter		Example
1	Calibration Sample Name	up to 8 letters	SAMPLE
2	Sample Time	1 - 300 seconds	15
3	Delay Time	0.1 - 4.0 seconds	1
4	Desorption Time	0.1 - 4.0 seconds	4
5	Inhibit Time	10 - 999 seconds	50
6	Oven Temperature	30° C - 140° C	50
7	Chart Duration	1; 3; 5; 10; 15; 20; 30 minutes	30
8	Analysis per Calibration	1 - 99	99
9	Auto Analysis Duration	0 - 120 minutes	manual
10	Backflush Option	0 = off; 1 = on	
11	Detector	.... 1-AID, 2-ECD, 3-TCD, F-PID	AID
12	Column	up to 8 letters	6 ft. 10% CP5
13	Column Pressure	5 - 40	20
14	Number of Calibration Peaks	1 - 16	1
15	Peak Number 1 • Substance Name • Concentration Range • Calibration Concentration • Peak Alarm Values	 up to 8 letters 0 = ppm; 1 = ppb 99.9 ppm; 9999ppb 0 - 99.99 ppm; 9999ppb	 U1/AID PPM 1.00 99
16	Upload Scentograph Parameters		

1.10 DATA VALIDATION

As mentioned previously, peak identification is by retention time index (RTI). Sample spikes, using known levels of target compounds, can be prepared to identify the absence/presence of target compounds in the samples, if peaks are eluting close to the target compounds. Typically, only the RTI is needed to identify the peaks of interest. Quantification is determined from the linear calibration curve, and solving for concentration "(y)" from the straight line equation. The coefficient of variation on the straight line equation should have an R squared (R^2) of 0.95 or better. Confirming the identity of any particular target compound must be done by other analytical methods, typically GC/MS. Standards must be run along with the samples and should bracket the levels found in the field samples.

Alternatively, a statistical approach to data validation can be sought. Once the linear range is established, an appropriate standard of either low or midrange concentration will be analyzed 10 or more times throughout the day. The standard deviation of the mean ($\sigma(n-1)$) for the response of the standard selected is determined. The statistical method detection limit (MDL) will be three times the standard deviation (3σ). The method quantitation limit (MQL) will then be 10 times the standard deviation (10σ). Results below the MDL are considered "nondetects" (ND). Results above

the MDL but below the MQL are considered "detected", but below the quantitation limit, so are ascribed a "J" value. This "J" value will flag the data to let the user know the results are questionable. Results above the MQL are considered statistically reliable data.

1.11 HEALTH AND SAFETY

Analysis should be performed in a well-ventilated room. When liquid reagents are used to prepare standards, etc., disposable protective gloves and suitable eye protection should be worn. Work should be performed under a vented hood.

2.0 PORTABLE XRF ANALYZER: SOP #1707

2.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for portable X-ray fluorescence spectroscopy (XRF), an analytical technique which allows for qualitative and quantitative analysis of a sample's chemical composition. In a source-excited XRF analysis, primary X-rays emitted from a sealed radioisotope source are utilized to irradiate a sample. These X-rays cause the sample to emit characteristic fluorescent X-rays from the elements contained in the sample. From the energy, or wavelength, of these fluorescent X-rays, a qualitative analysis can be made. From the number of X-rays at a given energy, a quantitative analysis is possible. Solid and liquid samples can be analyzed with proper X-ray source selection. Typical environmental applications are:

- heavy metals in soils, sludges, and liquids;
- light elements in liquids;
- heavy metals in industrial waste stream effluent;
- PCBs in transformer oil by Cl analysis; and
- heavy metal air particulates collected on membrane filters.

Measurements may be made in situ, or samples may be collected, homogenized, and placed into sample cups for analysis.

2.2 METHOD SUMMARY

XRF instruments use radioactive isotopes, such as Fe-55, Cm-244, Cd-109 and Am-241, for the production of primary X-rays. Each source emits a specific energy range of primary X-rays that causes a corresponding range of elements in a sample to produce fluorescent X-rays. When more than one source excites the elements of interest, the appropriate source(s) is selected according to its excitation efficiency for the elements of interest.

For measurement, the sample is positioned in front of the source-detector window and exposed to the primary (source) X-rays by pulling a trigger on the probe (or pushing back the top of the probe unit on the sample type probe), which exposes the sample to radiation from the source. The sample's fluorescent and backscattered X-rays enter through the detector beryllium (Be) window and are detected in the active volume of a high-resolution, gas-filled, proportional counter.

Elemental count rates (the number of net element pulses per second) are used in correlation with actual sample compositions to generate calibration models for qualitative and quantitative measurements.

The user selects an analysis time from 1 to 32,767 seconds. Generally, the shorter measurement times (30 s - 100 s) are used for initial screening and hot spot delineation, while longer measurement times (100 s - 500 s) are used for higher precision and accuracy requirements.

2.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Use appropriate sample containers (glassware) with Teflon-lined lids for sample collection. Disposable 31-mm diameter plastic cups are used for XRF measurements.

2.4 INTERFERENCES AND POTENTIAL PROBLEMS

- **Sample Placement.** The X-ray signal decreases the greater the distance from the radioactive source. Minimize decrease by maintaining the sample's distance from the source within a 1-mm range.
- **Sample Representation.** Representation is affected by the soil macro- and micro-homogeneity. This can be minimized by either homogenizing large samples prior to analyzing an aliquot, or by analyzing several samples (in

situ) at each sampling point and averaging the results.

- **Reference Analysis.** XRF soil chemical and physical matrix effects may be corrected by using inductively coupled plasma (ICP), or atomic absorption (AA) spectrometer-analyzed, site-specific soil samples as calibration samples. A major source of error can result if the samples analyzed are not representative of the site and/or the analytical error is large. With XRF calibrations based on reference analysis results, the XRF analytical results may be in the same units used for the calibration samples reference analysis. For example, total metals can be used for comparison using the toxicity characteristic leachate extraction procedure (TCLP).

- **Chemical Matrix Effects.** Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. It is critical to establish all chemical matrix relationships during the time of instrument calibration. This is done by using ICP or AA analyzed soil samples as the XRF calibration standard (as discussed in Step 3).

- **Physical Matrix Effects.** Physical matrix effects result from variations in the physical character of the sample. They include such parameters as particle size, uniformity, homogeneity and surface condition. Physical matrix effects can be minimized by sieving and thoroughly homogenizing a soil sample prior to analysis.

- **Ambient Temperature.** Changes in ambient temperature can affect the gain of the amplifiers producing instrument drift. As long as the gain control is allowed to make periodic adjustments however, the unit will compensate for the influence of temperature on its energy scale. If increasing or decreasing ambient temperature is a concern, allow the XRF unit to gain control after every five measurements.

- **Moisture Content.** Moisture content of soil samples poses potential interference. Soil samples should have a percent moisture of no more than 20%. Dry samples, if necessary.

- **X-Ray Spectrum Overlap.** Certain X-ray lines from different elements (when present in the sample) can be very close in energy, and therefore, interfere by producing a severely overlapped spectrum. The lead/arsenic overlap is a typical example.

- **Pure Element Calibration.** When doing the pure element calibration, be sure to include all elements of interest at the site, as well as elements adjacent to the target compound. In addition to target elements, include all elements present in detectable amounts with the X-ray source being used. Use that information to determine potential spectral interferential effect on the target element.

2.5 EQUIPMENT/APPARATUS

- portable XRF-X-MET model 840, model 880 or equivalent
- pure element samples
- battery charger
- battery pack
- 31-mm diameter disposable sample cups
- polypropylene film, 0.2 mil thickness
- plastic bags
- 20-mesh sieve
- moisture balance oven
- nylon reinforced, water-repellant backpack
- metal reinforced shipping case with die-cut foam inserts

2.6 REAGENTS

- pure element spectral calibration standards
- matrix calibration standards -- ICP- or AA-analyzed site matrix calibration standards

2.7 PROCEDURES

2.7.1 Calibration

Calibration of the XRF should be based on previously collected samples which were analyzed by AA or ICP. The field-portable unit uses samples collected from the site to generate a calibration curve.

Samples used for XRF calibration, more commonly referred to as site-specific calibration standards

(SSCS), must be representative of the matrix and concentration range which will be sampled at the site. For example, if conducting an investigation for off-site contamination, then SSCS samples should not come from on site. The matrices could be different and may introduce a source of error in the calibration model.

In addition, a full concentration range of the target element(s) of interest is needed to generate a representative calibration curve. The highest and lowest SSCS samples will be used to determine the linear calibration range. Samples used to generate the calibration curve must be prepared in a fashion similar to samples analyzed by the XRF. Samples used for the calibration, especially those samples at approximately the level of concern, are used as QC check samples during field activities.

The more the sampler knows about how the matrix varies at the site, the more representative the calibration model, and the more accurate the results. A minimum of 10 samples are recommended to generate the calibration; the maximum number is 30. A general rule is to add 5 samples for each element of interest and 5 samples for each adjacent element. As the number of elements under analysis increases, more calibration samples are required to adequately characterize the concentration ranges present for each element. Be aware that there may be a need for more than one calibration model to maintain linearity over the concentration ranges in question.

2.7.2 In Situ Soil Analysis

In situ soil measurements provide a rapid means of data collection at a large number of sample points, eliminate the need for sample containers and chain of custody forms, and yield real-time measurements. Two primary scenarios exist for in situ measurements. First, perform rough, rapid, non-quantitative XRF screening with a non-site-specific or generic calibration model; this allows for decisions such as where to collect samples for use in a site-specific calibration model. This scenario takes into account previous data collected at the site, a visual inspection of the site, and historical background information. Care should be exercised when using non-site-specific or generic calibration models, as variations in sample representativeness and matrices may significantly limit the validity of the model. Second, in situ XRF screening is conducted with a site-specific calibration model providing rapid, semi-quantitative data.

In addition to these two in situ scenarios, XRF analysis may be performed on samples which have been collected and undergo some type of sample preparation. When sampling is employed, the prepared sample is analyzed by XRF measurement, and the same portion is then submitted for confirmation analysis by AA/ICP, according to QA/QC protocols. A split portion of the sample is archived.

Remove all surface debris in the sampling location. To avoid cross contamination, place a single thickness plastic sample bag or polypropylene film over the probe and firmly press the probe to the ground. Where possible, maximize instrument performance by placing it 3 mm from the ground surface. It should be noted that in situ measurements can exhibit a high degree of variability due to the natural heterogeneity of the soil. This effect may be minimized by averaging multiple readings within a predetermined area based on sampling objectives. Single in situ measurements may be used at any one sample location; however, this can reduce the precision and representativeness associated with the XRF results. Therefore, the minimum recommended measurement time or duration for in situ analysis is 30 seconds. All sample readings should be recorded.

The depth of X-ray penetration for analysis is only the first two or three millimeters of soil. Therefore, **in situ measurements represent data from the ground surface only, and in no way reflect sub-surface soil conditions.** As little as 0.5 cm of clean soil cover can completely mask a hot spot. Also, in situ analysis can have a high degree of error due to the heterogeneity and possible wide range of particle size fractions present in the sample. Data obtained from in situ analysis is best suited for site characterization activities where the user is interested in obtaining a quick overview of site conditions at the surface. It can also define extent of contamination if confirmation samples are used.

2.7.3 Soil Samples for XRF Analysis

Discrete sample collection involves physical collection of a soil sample (4 oz.) and some type of field preparation prior to XRF analysis. Two types of discrete XRF sampling are utilized in the field. One type involves collecting a sample in a plastic bag, rough sieving it to remove organic debris and rocks, and homogenizing it by mixing in the bag.

Take the XRF reading by directly measuring through the sample bag. Take three measurements from each bag, shaking the bag between measurements. A measurement time of 15-30 seconds is recommended. Be consistent with sampling time for all samples collected. Once XRF measurements are complete, prepare a minimum of 10% of the samples for confirmation analysis by AA/ICP. It is imperative that the same sample utilized for XRF analysis be sent for the confirmation analysis; this is typically accomplished by splitting the volume of the original sample prepared, one half being sent for analysis to the lab and one half being archived.

A second method of discrete XRF sampling involves a more rigorous method of sample preparation. The first step in sample preparation is drying, either by air, in a conventional oven at 105°C, or with a moisture balance. Drying is a recommended step and is necessary based on moisture content of the soil. Use care when using a microwave oven or conventional oven for drying due to their potential to vaporize lead, arsenic and mercury, if present in samples. Not only does this lead to error, but it may be a health hazard. Small, air-circulating or moisture balance ovens are recommended. Proper respiratory protection should be worn.

Once dry, remove any visible organic debris and sieve the sample through a 20-mesh sieve. Fill a sample cup with the prepared sample and retain it for confirmation analysis. Settle contents in the sample cup by tapping; this will yield a more smooth, uniform surface for analysis. Cover the sample with polypropylene film (making certain there are no wrinkles in the film) and analyze it for 240 seconds. Shake the cup and analyze again. Repeat this procedure for a total of three readings on each sample. Record the individual results and calculate an average. Save the prepared sample cup. A minimum of 10% of the prepared sample cups should be sent for confirmational analysis by AA/ICP procedures.

2.8 CALCULATIONS

Mathematically model the matrix calibration for optimum linearity, as per the XRF operating instructions.

2.9 QUALITY ASSURANCE/ QUALITY CONTROL

There are a number of QA/QC measurements which must be utilized when performing XRF analysis.

- **Precision** is determined by repeated measurements of a low matrix calibration standard at the beginning of sampling activities, and then after every tenth sample. This sample is analyzed for the same time as the field samples. The precision objective for XRF should be ±20% relative standard deviation. A mid-range matrix calibration sample is also recommended to aid in determining instrument precision. This sample should be at or near the concentration level of interest. By running low-level calibration samples, gain changes and baseline drift can be monitored.

- **Accuracy** is best determined by using site-specific, low-, mid- and high-level calibration samples that are analyzed by AA/ICP.

- **Representativeness** is best determined by developing an adequate sampling scheme which characterizes the range of contaminants and matrix variability at the site. When the calibration samples accurately characterize the range of contaminant concentrations on site, the calibration model will be representative of the site.

- The question of **comparability** arises when XRF data are compared to AA or ICP data obtained from a sample digestion procedure. XRF data may not be comparable to data obtained by AA or ICP. The portable XRF should be used as a screening tool in conjunction with these more rigorous analytical methods, especially with the acid digestion method procedures. Using AA/ICP, samples should be prepared in the same manner as those for XRF analysis.

- **Completeness** is determined on a site-specific basis and is a measure of the desired number of samples analyzed versus the actual number of samples analyzed.

- **Replicates** are recommended at a minimum rate of 5-10%. Replicate samples should be prepared independently of other samples and go through the same sample preparation procedure. Replicates are a check on homogeneity of the sample matrix, consistency of sample preparation, and precision of the analysis.

- **Confirmation samples** are recommended at a minimum rate of 10%. Ideally, the sample that was analyzed by XRF should be the same sample that is sent for AA/ICP confirmation analysis. When confirming an in situ analysis, collect a sample from a six-inch by six-inch area for both an XRF measurement and a confirmation analysis. The correlation factor between XRF and AA/ICP data should be 0.7 or greater.

- **Performance evaluation (PE) samples** are another possible QC mechanism for checking AA/ICP analysis, but are not typically applicable to XRF analysis due to a dissimilar matrix. If the site matrix does not match the PE matrix, the user should not utilize the PE samples.

1.10 DATA VALIDATION

This section is not applicable to this SOP.

1.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific health and safety procedures.

3.0 PHOTOIONIZATION DETECTOR (HNU): SOP #2056

3.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the procedure for using a photoionization detector (PID). The PID is a portable, non-specific, vapor/gas detector employing the principle of photoionization to detect a variety of chemical compounds, both organic and inorganic, in air. This procedure is applicable to the HNU-PI 101.

3.2 METHOD SUMMARY

The PID is a useful general survey instrument at hazardous waste sites. A PID detects and measures real-time concentrations of many organic and inorganic vapors in air. A PID is similar to a flame ionization detector (FID) in application; however, the PID has somewhat broader capabilities as it can detect certain inorganic vapors. Conversely, the PID is unable to respond to certain low molecular weight hydrocarbons, such as methane and ethane, which are readily detected by FID instruments.

The PID employs the principal of photoionization. The analyzer responds to most vapors with an ionization potential less than, or equal to, that supplied by the detector, which is an ultraviolet (UV) lamp. Photoionization occurs when an atom or molecule absorbs a photon of sufficient energy to release an electron and form a positive ion. This action occurs when the ionization potential of the molecule in electron volts (eV) is less than the energy of the photon. The sensor is housed in a probe and consists of a sealed ultraviolet light source that emits photons with an energy level high enough to ionize many trace organics, but not enough to ionize the major components of air. A chamber exposed to the light source contains a pair of electrodes, one bias electrode and one collector electrode. When a positive potential is applied to the bias electrode, a field is created in the chamber. Ions formed by the adsorption of photons are driven to the collector electrode. The current produced is then measured and the corresponding concentration is directly displayed on a meter in units above background. Several probes are available for the PID, each having a different source and a different ionization potential. The selection of the appropriate probe is essential to obtain useful field results. Though it can be calibrated to a particular compound, the instrument cannot distinguish between detectable compounds in a mixture of gases, and therefore produces an integrated response to the mixture.

Three probes, each containing a different UV light source, can be used with the HNU. Energies are 9.5, 10.2, and 11.7 electron volts (eV). All three detect many aromatic and large molecule hydrocarbons. In addition, the 10.2 eV and 11.7 eV probes detect some smaller organic molecules and some halogenated hydrocarbons. The 10.2 eV probe is the most useful for environmental response work, as it is more durable than the 11.7 eV probe and detects more compounds than the 9.5 eV probe.

Gases with ionization potentials near to, or less than, that of the lamp will be ionized, and thus be detected and measured by the analyzer. Gases with an ionization potential higher than that of the lamp will not be detected. The ionization potentials for various atoms, molecules, and compounds are given in Appendix B. The ionization potential of the major components of air (oxygen, nitrogen, and carbon dioxide) range from about 12.0 eV to about 15.6 eV, so are not ionized by any of the three lamps.

Ionization sensitivity for a number of chemical groupings when exposed to photons from a 10.2 eV lamp is illustrated in Table 2. Applications of each probe are included in Table 3.

While the HNU is primarily used as a quantitative instrument, it can also be used to detect certain contaminants, or at least narrow the range of possibilities. Noting instrument response to a contaminant source with different probes eliminates some contaminants from consideration. For instance, a compound's ionization potential may be such that the 9.5 eV probe produces no response, but the 10.2 eV and 11.7 eV probes do elicit a response.

Table 2: Relative Photoionization Sensitivities for Various Gases

Chemical Group	Relative Sensitivity	Examples
Aromatic	10	benzene, toluene, styrene
Aliphatic Amine	10	diethylamine
Chlorinated Unsaturated	5 - 9	vinyl chloride, vinylidene chloride, trichloroethylene
Carbonyl	5 - 7	MEK, MIBK, acetone, cyclohexanone
Unsaturated	3 - 5	acrolein, propylene, cyclohexanone, allyl alcohol
Sulfide	3 - 5	hydrogen sulfide, methyl mercaptan
Paraffin (C_5-C_7)	1 - 3	pentane, hexane, heptane
Ammonia	0.3	
Paraffin (C_1-C_4)	0	methane, ethane

Note: Relative sensitivity is the actual HNU meter reading observed when measuring a 10 ppm gas concentration of the listed chemical, using the 10.2 eV probe (calibrated for 10 ppm of benzene) and a span setting of 9.8 (for direct reading of benzene).

3.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

This section is not applicable to this SOP.

3.4 INTERFERENCES AND POTENTIAL PROBLEMS

3.4.1 PID Instrument Limitations

- The PID is a non-specific, total vapor detector. It cannot be used to identify unknown substances; it can only quantify them.
- The PID must be calibrated to respond to benzene using isobutylene calibration gas which has been analyzed and compared to a known benzene standard.
- The PID does not respond to certain low molecular weight hydrocarbons, such as methane and ethane, and does not detect a compound if the probe has a lower energy than the compound's ionization potential. Methane absorbs ultraviolet lamp energy. One-half percent methane will result in a 30 percent reduction in detector sensitivity and 5 percent methane will yield a 90 percent decrease in detector sensitivity.
- Certain toxic gases and vapors, such as carbon tetrachloride and hydrogen cyanide, have high ionization potentials and cannot be detected with a PID.
- Certain models of PID instruments are not intrinsically safe. The HNU-PI 101 PID is not designed for continuous use in potentially flammable or combustible atmospheres. Therefore, a PID should be used in conjunction with a Combustible Gas Indicator.

Table 3: Typical Applications of Interchangeable Probes

Compound	Ionization Potential (eV)	Relative Sensitivity	
p-xylene	8.44	0.10	0.104
p-chlorotoluene	8.70	0.09	0.112
toluene	8.82	0.09	0.112
o-chlorotoluene	8.83	0.075	0.112
ethyl acetate	9.19	0.075	0.112
benzene	9.24	0.10	0.10
methyl mercaptan	9.24	0.10	0.072
pyridine	9.32	0.075	0.122
allyl alcohol	9.67	0.10	0.112
crotonaldehyde	9.88	0.075	0.104
amyl alcohol	9.80	0.09	0.116
cyclohexane	9.88	0.075	0.104
vinyl chloride	9.95	0.085	0.112
butanol	10.94	0.09	0.176
ammonia	10.15	0.06	0.160
acetic acid	10.37	0.04	0.560
ethylene	10.52	0.0	0.320
ethylene oxide	10.56	0.0	0.298

Note: Relative sensitivity equals the response with the 9.5 eV probe or the 11.7 eV probe divided by the response with the 10.2 eV probe.

- Electrical power lines, radiotransmissions or power transformers may cause interference with the instrument and thus cause measurement errors.
- Winds and high humidity will affect measurement readings. The HNU may become unusable under foggy or humid conditions, when condensation occurs on the lamp. This is indicated by the needle dropping below zero, or a slow constant climb on the read-out dial.
- The lamp window must be periodically cleaned to ensure the maximum transmission of the ionizing protons into the detector chamber.
- The 11.7 eV lamp window is a type of fluoride crystal which is hygroscopic and

will absorb water vapor from the sample stream. In high humidity applications, the lamp should be cleaned with an oil-free, chlorinated hydrocarbon, or freon on a daily basis.

- The HNU measures concentrations from about 1-2000 ppm, although the response is not linear over this entire range. For example, the response to benzene is linear from about 0-600 units above background. This means the HNU reads a true concentration of benzene only between 0 and 600. Greater concentrations are detected at a lower level than the true value.

- Do not use this instrument for head space analysis where liquids can inadvertently be drawn into the probe.

3.4.2 Regulatory Limitations

- Transport of calibration gas cylinders by passenger and cargo aircraft must comply with the U.S. Code of Federal Regulations, 49 CFR Parts 100-177. A typical calibration gas included with a PID is isobutylene. It is classified as a non-flammable gas, UN #1556 and the proper shipping name is Compressed Gas. It must be shipped by cargo aircraft only.

3.5 EQUIPMENT/APPARATUS

- PID (HNU)
- operating manual
- probes: 9.5 eV, 10.2 eV, or 11.7 eV
- battery charger for PID
- spare batteries
- jeweler's screwdriver for adjustments
- Tygon tubing
- NBS traceable calibration gas (type)
- "T" valve for calibration
- field data forms
- intake assembly extension
- strap for carrying PID
- Teflon tubing for downhole measurements
- plastic bags for protecting the PID from moisture and dirt

Note: This instrument may be kept on continuous charge without battery damage.

3.6 REAGENTS

- isobutylene standards for calibration
- methanol for cleaning ionization chamber (GC grade)
- mild soap solution for cleaning unit surfaces
- specific gas standards when calibrating to a specific compound
- light source cleaning compound Cat No. PA101534-A1 (for 9.5 and 10.2 eV lamps only)
- oil-free, chlorinated solvent, or freon for cleaning the 11.7 eV lamp

The HNU is calibrated according to the operations manual, using isobutylene as the calibration standard. Also, refer to the operations manual for alternate calibration to a specific compound. Current HNU manuals contain an error with the isobutylene response factor for the 10.2 eV lamp. The manual indicates 7.0 whereas the correct value is approximately 5.5.

3.7 PROCEDURES

3.7.1 Start-up

1. Check to ensure the proper operation of the PID, as appropriate, using the equipment checklist provided in Sections 3.5 and 3.6 and the steps listed below.

2. Allow the temperature of the unit to equilibrate to its surrounding (about 5 minutes).

3. Attach the probe to the read out unit. Match the alignment key, then twist the connector clockwise until a distinct locking is felt.

4. Turn the *Function* switch to the battery check position. Check to ensure that the indicator reads within or beyond the green battery arc on the scale plate. If the indicator is below the green arc, or if the red LED comes on, the battery must be charged prior to using.

5. To zero the instrument, turn the *Function* switch to the Standby position and rotate the *Zero Potentiometer* until the meter reads zero. Wait 15-20 seconds to ensure that the zero adjustment is stable. If not, then readjust.

6. Check to see that the *Span Potentiometer* is set at the appropriate setting for the probe being used.

7. Set the *Function* switch to the desired range.

8. Listen for the fan operation to verify fan function.

9. Look for ultraviolet light source in the probe to verify function. Do not look at this light source from closer than 6 inches with unprotected eyes; observe briefly.

10. Prior to survey, check instrument with an organic point source such as a Magic Marker to verify instrument function.

11. Routinely throughout the day, verify the remaining useful battery life by turning the *Function* switch to BATT. Schedule the instrument's use accordingly.

3.7.2 Field Operation

Field Calibration

1. Follow the startup procedure in Section 3.7.1.

2. If the PID does not start up, check out, or calibrate properly, the instrument should not be used. Under no circumstances should work requiring PID air monitoring be done without a properly functioning instrument.

3. Set the *Function* switch to the range setting for the concentration of the calibration gas.

4. Attach a regulator to a disposable cylinder of calibration gas. Connect the regulator to the probe of the HNU with a piece of clean Tygon tubing. Open the valve on the regulator.

5. The latest technical information from HNU is that the external *Span Adjustment Control* should be used to calibrate the instrument. That is, if you are calibrating an HNU to a 60 ppm isobutylene as benzene standard with the unit set at 9.8, and the HNU reads 50 ppm, the external *Span Adjustment Control* should be adjusted to a lower number setting until the correct reading has been obtained. The lower the number on the Span Adjustment Control the greater the instrument sensitivity.

6. Record the following information in the site logbook: the instrument ID number (EPA decal or serial number if the instrument is a rental), the initial and final span settings, the date and time, concentration and type of calibration gas used, and the name of the person who calibrated the instrument.

7. Record the calibration data in the field.

8. In some field applications, with the exception of the probe's inlet and exhaust, the PID should be wrapped in clear plastic to prevent it from becoming contaminated and to prevent water from getting inside in the event of precipitation.

Operation

1. Record all readings in the site logbook. Readings should be recorded as "units above background," not ppm.

2. As with any field instrument, accurate results depend on the operator being completely familiar with the operator's manual. In order to obtain accurate results, follow the instructions in the operating manual explicitly.

3. Position the probe assembly close to the area to be monitored because the low sampling rate allows for only very localized readings. Under no circumstances should the probe tip assembly be immersed in fluid.

4. While taking care not to expose the PID to excessive moisture, dirt, or contamination, monitor the work activity as specified in the site health and safety plan. Conduct the PID survey at a slow to moderate rate and the intake assembly (the probe) should slowly sweep from side to side. There is a 3 to 5

second delay in read-out, depending upon the instrument's sensitivity to the contaminant.

5. During drilling activities, perform PID monitoring at regular intervals downhole, at the headspace, and in the breathing zone. In cases with elevated organic vapor levels, monitor in the breathing zone during actual drilling. When the activity being monitored is not drilling, readings should emphasize breathing zone conditions.

6. When the activity is completed, or at the end of the day, carefully clean the outside of the PID with a damp disposable towel to remove any visible dirt. Check calibration again before storing. Return the PID to a secure area and place on charge.

3.7.3 Post Operation

1. Turn *Function* switch to "off."

2. Place the instrument on the charger. When on charge, the probe must be connected to the readout unit to ensure charging.

3. Complete logbook entries, verify the accuracy of entries, and sign/initial all pages. Following completion of a series of "0" readings, verify the instrument is working.

4. Check the equipment, repair or replace damaged equipment, and charge the batteries.

3.8 CALCULATIONS

The HNU is a direct reading instrument. Readings are interpreted as units above background, rather than ppm.

3.9 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance activities which apply to the implementation of these procedures. However, the following QA procedures apply:

- All data must be documented on field data sheets or within site logbooks.

- All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and must be documented.

3.10 DATA VALIDATION

This section is not applicable to this SOP.

3.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific health and safety practices.

The HNU-PI 101 is certified for use in Class 1, Division 2, Groups A, B, C, and D.

4.0 PHOTOVAC 10A10 PORTABLE GAS CHROMATOGRAPH OPERATION: SOP #2107

4.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the use, calibration, and maintenance of the Photovac 10A10 portable gas chromatograph. The Photovac 10A10 gas chromatograph is used for field and laboratory analysis of air, soil gas, and water/soil headspace samples. Chlorinated and non-chlorinated alkenes and aromatic hydrocarbons down to the 1 to 20 part per billion (ppb) range can be detected.

4.2 METHOD SUMMARY

The Photovac 10A10 is a battery/AC-operated photoionization detector (PID) portable gas chromatograph. It is a field instrument capable of monitoring for many organic vapors using an ultraviolet light source and a photoionization detector. The samples are introduced into the 10A10 via gas-tight syringes. Gaseous contaminants are ionized as they emerge from the column. The ions are then attracted to an oppositely charged electrode, which causes a current and sends an electronic signal to a strip chart recorder or, alternately, to an integrator/plotter system.

4.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

This section is not applicable to this SOP.

4.4 INTERFERENCES AND POTENTIAL PROBLEMS

- This instrument should not be exposed to precipitation or high humidity.
- The instrument works best in a stable, temperature-controlled environment.
- Liquids should not be injected into this instrument.
- Readings can only be reported relative to retention times of the calibration standard used.
- Combustion fumes can contaminate the columns.
- High concentrations of short chain alkanes and alkenes in samples may interfere with the resolution and detector sensitivity of early-eluting chlorinated alkenes, and aromatic compounds.
- Since the Photovac is a GC, the target compounds are identified by their retention times (RTs). If the RT of the sample peak(s) matches the RT of the standard peak(s), they are assumed to be identical. If any non-target compound has the same RT, it can be misidentified as a target compound.

4.5 EQUIPMENT/APPARATUS

- Photovac 10A10 gas chromatograph, with manual and power cord
- extra source lamp
- Photovac lamp tuning screwdriver
- extra columns/fittings
- ultrazero air carrier gas
- two-stage regulator, with quick-connect fitting
- 1 flowmeter per Photovac, either bubble-meter, rotameter, or Gilibrator
- septa, 6 mm diameter
- syringes, gas-tight, 10 µL to 1 mL
- VOA vials filled with activated charcoal, for syringe cleaning
- integrator or strip-chart recorder, with appropriate connections
- labels
- extra Photovac integrator pens
- extra Photovac integrator paper
- tools – large adjustable wrench, wrenches (5/16 inch to 9/16 inch), screwdrivers (flat and Phillips head), needle-nose pliers, jeweler's screwdrivers, Allen wrenches
- duct tape

- Teflon tape
- power strip
- snoop
- Kimwipes (or similar lint/static free wipe)
- Pelican cases (or equivalent)

4.6 REAGENTS

- carrier gas cylinder (compressed ultrazero air, 0.1 ppm total hydrocarbons).
- headspace calibration standards (Supelco A and B or equivalent).
- certified gas calibration standards with a ± 2% level of accuracy (from Scott Speciality Gas, Matheson Gas, or other reliable source).

4.7 PROCEDURES

4.7.1 Laboratory Operation

1. Attach the carrier gas (air) cylinder to the 10A10, and, via the second stage of the dual-stage regulator, deliver a maximum of 40 psi.
2. The flow rate will vary according to the target compounds in question and the column used. Adjust the carrier gas flow using the knurled knob to the left of the 10A10, labeled Column 1 or Column 2. The flow is measured by attaching a flowmeter to the vent port at the top left of the unit. Once the flow is set, the PID will stabilize after approximately 1/2 hour of warm up time. Set the output at 10 mV using the offset knob in the center of the unit.
3. Attach an interface cable from the output lead on the 10A10 to a strip chart recorder, or preferably, a plotter-integrator such as the Hewlett Packard 3396A. The voltage input and/or attenuation is selected on the chart or integrator to keep peaks on scale. Check that the electrical controls are set as follows:
   - power switch to "off"
   - charge switch to "off"
   - attenuation switch to 100 (lowest sensitivity)
   - offset dial to zero
   - chart recorder connected to the coaxial output connector
   - chart recorder set to 100 mV (full scale) and chart speed to 1 cm/min
   - power cord plugged into the panel socket (the red AC indicator light will come on)

   The instrument is now in its power down condition and is ready for starting.
4. With the chart recorder off, switch on the power switch. The red *Source Off* indicator will light and stay on for up to 5 minutes. During this time the lamp-start sequence is being automatically initiated.
5. As soon as the *Source Off* light is extinguished, the meter shows a high reading which should fall as conditions in the photoionizing chamber stabilize.
6. Establish an acceptable base line on the chart recorder.

The instrument is now ready for calibration.

4.7.2 Calibration

Refer to Table B, Appendix B, for the calibration and maintenance schedule. Photovac Incorporated conducts an instrument calibration and includes the chromatogram as a component of that instrument's instruction manual. A check of the instrument's performance can be accomplished by duplicating the factory calibration check and comparing the results. The procedure is as follows:

1. Completely flush a clean 1-L sample bottle (or a clean 1-L Tedlar bag fitted with a septum cap) with good quality bottled air.
2. Using the factory calibration data sheet, calculate the required amounts of each calibration compound required to generate an air standard (with a total volume of 1 liter) which is identical to that run by Photovac in the factory calibration.
3. Using an appropriate volume gas-tight syringe, aspirate the required amount of each

compound from the headspace of the storage bottles at room temperature, and inject it into the purged 1-L sample bottle (or Tedlar bag). Be careful to fully flush the syringe with clean air before injecting a new compound.

4. Allow 10 minutes for the standard to equilibrate.

5. Using a clean 100 µl gas-tight syringe, aspirate the required injection volume from the 1-L standard. With a crisp and snappy action, inject the standard into the proper "injection port" of the Photovac 10A10.

6. Start and mark the strip chart recorder. The resulting chromatogram should be similar to the factory calibration chromatogram, under similar conditions.

7. A simple calibration curve can be constructed by injecting the same volume of several standards with varying concentration levels of the target compounds. Alternatively, a calibration curve can also be constructed by injecting various volumes (10 - 1000 µL) of the same standard. In this case, the response of the standards and samples should be normalized to one injection volume. Both standards and samples present in Tedlar bags can be diluted in the field.

4.7.3 Field Operation

1. Prior to any field analyses, check to ensure that the instrument is operational and clean. Remove closure fittings on the *Detector Out* port. Closure fittings may have been engaged to prevent static contamination.

2. Check that the carrier gas supply is adequate (charge supply is 1800 PSI and should last up to approximately 3 days, depending on carrier flow rates).

3. Set the pressure regulator to zero (fully counterclockwise) and turn on the main valve of the lecture bottle.

4. Slowly turn the regulator control clockwise until air begins to escape from the quick-disconnect connection. Allow the line to purge for 10 seconds.

5. Plug the quick-disconnect fitting into the free *Carrier in* port. Shut off and disconnect the laboratory air supply. Adjust the lecture bottle regulator to 40 psig. Set the required flow rate as described previously using a bubble meter, calibrated rotameter, or Gilibrator.

6. With the instrument in the "power down" mode, disconnect the AC power supply. This automatically switches the instrument to battery power. The instrument is now completely self-contained, and, with a battery powered recorder, may be taken into the field. Check the battery charge on the Photovac.

7. The instrument is now ready to be run through the start-up procedures described under Laboratory Operation, parts 4-8 of the manual.

8. If there is a significant change in ambient temperature when the instrument is moved from one place to another, the column will require time to stabilize thermally. At higher sensitivities, a non-thermally stabilized column will manifest itself as baseline drift.

9. For troubleshooting information, refer to Appendix B.

4.7.4 Shut Down

1. Turn the power switch to "off."

2. Reduce the carrier gas flow to 2-5 cm^3/min.

3. Place the instrument on low charge while on the bench and maintain it as described in Table F and Section 4.7.6 below.

4. Unplug the unit except when charging batteries.

4.8 CALCULATIONS

4.8.1 Calibration Curve

A calibration curve of at least three concentrations must be constructed for each target compound. A straight line equation in the form of $y = (m)(x)+b$ (where: x = concentration, y = area counts, m = slope and b = the intercept) is fit to the standards raw data. The (y), or the unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative

of detector response range limitations.

Alternatively, sample concentration can be calculated as shown below:

$$[Sample] = [Std] \frac{A_1}{A_2} \cdot \frac{V_2}{V_1}$$

where:

sample	=	concentration of sample (ppb or ppm)
A_1	=	peak area of sample (volts x seconds)
A_2	=	peak area of standard (volts x seconds)
V_1	=	injection volume of sample (μL)
V_2	=	injection volume of standard (μL)
std	=	concentration (ppb or ppm)

4.8.2 Standard Response Generation/Duplication of Factory Calibration Data

If appropriate gas standard mixtures are not available, gas standards can be made using the headspace from 40-mL VOA bottles, with Teflon-lined septa screw caps, partially filled with the desired neat volatile liquid. Factory instrument response is generally determined using the following three compounds:

Compound	P_{vap} @ 20° C
methylene chloride	347 mm Hg
n-hexane	126 mm Hg
benzene	74 mm Hg

These compounds are toxic and should be stored and worked with under a hood. The general formula for preparing a standard from the headspace above a volatile liquid is:

$$V_{HS} = \frac{760\ (C)\ (V)}{P_{vap}}$$

where:

V_{HS}	=	volume of headspace (μL)
P_{VAP}	=	vapor pressure of liquid (mm HG)*
C	=	desired concentration (ppm)
V	=	volume of standard vessel (liters)

* Use appropriate tables to determine compound vapor pressure if working environment is not 20° C.

A determined volume of neat liquid headspace may be introduced to the standard vessel through the septa if using a Tedlar bag is used with the appropriate fitting. Bags or vessels used should be labelled with content concentrations, date, and time of preparation.

4.9 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance activities which apply to the operation of the Photovac. However, all instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling or operation and they must be documented.

4.10 DATA VALIDATION

This section is not applicable to this SOP.

4.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific health and safety practices.

5.0 PHOTOVAC 10S50, 10S55, AND 10S70 GAS CHROMATOGRAPH OPERATION: SOP #2108

5.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) pertains to the use, calibration, and maintenance of the Photovac 10S series portable gas chromatographs. The 10S series gas chromatographs are used for field and laboratory analysis of air, soil gas, and water and soil headspace. It tests for chlorinated and non-chlorinated, alkene and aromatic, compounds with detection limits of 1-5 ppb (parts per billion) for headspace analysis and 10-50 ppb for soil gas analysis.

5.2 METHOD SUMMARY

The Photovac 10S series are battery/AC operated, portable gas chromatographs with photoionization detectors. They are field/laboratory instruments capable of screening for many organic vapors using an ultraviolet light source and photoionization detector. Gaseous contaminants are ionized as they emerge from the column. The ions are then attracted to an oppositely charged electrode which causes a current and sends an electronic signal to the Photovac internal microprocessor or optional integration device. Refer to ERT SOP #2109, Photovac GC Analysis for Air, Soil Gas, Water, and Soil, for additional information.

5.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

This section is not applicable to this SOP.

5.4 INTERFERENCES AND POTENTIAL PROBLEMS

- These instruments should not be exposed to precipitation or high humidity.
- The instruments are best utilized in stable, temperature-controlled environments (even when using the internal temperature-controlled oven assembly, which may only reduce the effect of external atmospheric temperature variances). When used directly in the field, it is best to maintain constant temperatures to avoid the fluctuating retention times which may occur with changing temperatures.
- Readings can only be reported relative to retention times of the calibration standard used.
- Combustion fumes can contaminate the column.
- High concentrations of short chain alkanes and alkenes in samples may interfere with the resolution and detector sensitivity of early-eluting chlorinated alkenes and aromatic compounds.
- Since the Photovac is a GC, the target compounds are identified by their retention times (RTs). If the RT of the sample peak(s) matches the RT of the standard peak(s), they are assumed to be identical. If any non-target compound has the same RT, it can be misidentified as a target compound.

5.5 EQUIPMENT/APPARATUS

5.5.1 Equipment List

- Photovac 10S series gas chromatograph, with manual and power cord
- extra source lamp
- Photovac lamp tuning screwdriver
- extra columns/fittings
- ultrazero air carrier gas
- two-stage regulator, with quick-connect fitting
- one flowmeter per Photovac, either bubble-meter, rotameter, or Gilibrator
- septa, 6-mm diameter
- syringes, gas-tight, 10-μL to 1-mL
- VOA vials filled with activated charcoal, for syringe cleaning
- extra Photovac integrator pens

- extra Photovac integrator paper
- labels
- tools -- large adjustable wrench, wrenches (5/16 inch to 9/16 inch), screwdrivers (flat head and Phillips head), needle-nose pliers, jeweler's screwdrivers, Allen wrenches
- duct tape
- Teflon tape
- power strip
- snoop
- Kimwipes (or similar lint/static free wipes)
- Pelican cases

5.5.2 Carrier Gas Supply System Options

In most applications, the carrier gas used is ultra-zero compressed air (<0.1 ppm THC).

Use of the Internal Reservoir as Carrier Gas Supply

To recharge the internal reservoir, a special (optional) device known as the High Pressure Filling Station is needed. This device consists of the high pressure connection (normally male, left-hand thread) for cylinder attachment followed by a two-way valve. The two-way valve can be positioned to deliver gas from the cylinder to a long flexible hose and then to the *Refill* receptacle located on the rear right of the 10S50 panel. Alternatively, the valve can be turned to release high pressure gas held in the hose, after filling. A pressure relief valve is provided for safety at the upper end of the flexible hose. This is set at 1700 psi. A flow restricter is also incorporated in line with the hose, to limit the escape rate of gas. **Under no circumstances should the high pressure filling station be modified or disassembled by the user.** In the event of any problems, the unit must be returned to Photovac for repair or replacement.

Filling procedure is as follows:

1. Attach the high pressure fitting to the gas cylinder and turn the valve handle so that it points away from the cylinder.

2. Open the cylinder valve and check for leaks.

3. Now turn the filling station valve so that it points toward the cylinder. A steady flow of gas will be heard escaping from the end of the flexible hose; this purges the hose of any impurities.

4. Return the filling station valve to its previous position.

5. Place the Photovac on a sturdy, flat surface within easy reach of the flexible receptacle; press in firmly until a click is heard.

6. Turn the filling station valve until it again points toward the cylinder and carefully watch the *Contents* gauge on the Photovac as the needle climbs.

7. As the needle reaches approximately 1600 psi, the pressure relief valve on the high pressure filling station should prevent further increase. The pressure indicated on the *Contents* gauge must not exceed 1800 psi; switch the pressure relief valve before this occurs. Switching the valve allows high pressure gas trapped in the flexible hose to escape, while a check valve in the Photovac prevents the reservoir contents from escaping.

8. After relieving the pressure in the hose, it can be removed from the *Refill* receptacle by pulling upward on the knurled collar and extracting the fitting.

Use of External Tank for Carrier Gas Supply

Whenever possible, connect the Photovac to an external air cylinder to ensure a longer, more stable carrier gas supply. A high quality, two-stage regulator with output at 40 psi is required. Connection between this regulator and the Photovac is made using 1/8-inch Teflon tubing with a male quick-connect fitting attached to one end.

5.6 REAGENTS

- carrier gas, ultrazero air (<0.1 ppm total hydrocarbons)
- appropriate calibration standards (gas or liquid)

5.7 PROCEDURES

5.7.1 Shipping

The Photovac is initially shipped to the user in a cardboard box provided by Photovac International. Repeated shipping of Photovacs to field locations (in these boxes) has resulted in occasional electrical and mechanical problems. Shipping Photovacs in a more rugged container, such as the Pelican King Size Case (Orr Safety Equipment Co.), or equivalent is recommended. The Photovac can be left in this case while in use to prevent static electricity and to provide thermal stability.

5.7.2 Pre-Operational Checkout

1. Check the instrument for any obvious damage. Plug the power cord into an AC power source, if available. Remove any closure fittings which may have been affixed to the *Detector Out*, *Aux Out*, and *Cal Out* ports in order to minimize contamination.

2. Raise the computer module. Check that all compression fittings associated with the columns (the pre-column and analytical column) and all valves which are subject to carrier gas flow, are finger tight. **Do not over-tighten.** Fittings may loosen in transit. Check which injection port is connected to the columns (only one may be used at a time). Normally, the Photovac is supplied with an SE-30, 5% packed, 6-inch pre-column and 4-feet of analytical column unless otherwise specified (see Figure 1, Appendix B). If a capillary column (usually CP Sil-5, blue, equivalent to the properties of the SE-30) with oven temperature control module is being used, make sure the oven ribbon connection is tight. Attach the external 12V battery with adapter to the BNC connector (labeled *ext DC*) on the left side of the Photovac top panel. Adjust the oven module temperature to at least 5° greater (usually $\geq$ 40°C) than internal operating temperature. Close the computer module. Allow at least 40 minutes for oven temperature stabilization.

3. Check that the septum is new. Make sure the septum retainer is tight. **Do not overtighten.**

4. Engage the carrier gas flow (ultrazero air). Carrier gas can be introduced either by connecting an external low pressure source (attached by a Quick-Connect to the *External Carrier In* receptacle) or by recharging the Photovac internal high pressure refill attachment. If operating from an external cylinder, a clean, GC grade, two-stage regulator should be used. Set delivery pressure to a maximum of 40 psi. One-eighth inch Teflon lines, brass or stainless steel swagelock fittings and a quick-connect are used to attach the external carrier gas to the Photovac GC. Lines are purged 5-10 seconds with ultrazero air carrier gas before connection to the Photovac *External Carrier* gas inlet.

5.7.3 Carrier Gas Flow Rate Adjustment

1. Set carrier gas flow rates by attaching appropriate flow rate indicators (calibrated rotameters, Gilibrators, bubble meters, etc.) to the *Detector Out* port (red dial) and adjusting the appropriate needle valves to give a flow of 40-50 cm^3/min for a packed column and 10-15 cm^3/min for a capillary column.

2. After flow rate through the detector is set, turn on the instrument to warm it up while fine tuning the desired flow rates. For at least one hour prior to use, purge the instrument (column/valving/detectors) of residual contamination encountered in transit by carrier gas flow.

5.7.4 Photovac Settings

1. Press <ON> to turn on the instrument. "Lamp not ready" appears on the LCD. It takes approximately 1-2 minutes for the lamp to light. **Do not allow more than 3 minutes for the lamp to light, or electronic problems can occur.**

2. To enter the date and time, press <USE> in the *Library* section on the top control panel (there are four libraries which may be used). Then press <ENTER>. The LCD will prompt for entry of the "date" and "time."

3. Obtain a listing of compounds contained in the library selected for use by pressing <LIST>, then <ENTER>. If any compounds are not needed, they may be deleted by pressing <EDIT>. You will be prompted by the LCD

to enter the ID of the compound in the library you wish to edit, and the ID# as stated on the printout. Press <CLEAR>, then <ENTER>. That compound is then removed from the library. Repeat the edit sequence until all undesired compounds have been removed from the library selected for use.

4. Whenever the Photovac is turned off and then on, it reverts to the default gain setting of 2. A gain setting of at least 50 is necessary for detection of most common pollutants (i.e., aromatics and chlorinated alkenes) down to a 5-20 ppb range using a 100-250 μL sample injection volume. Since this is a non-destructive detector, small sample injection volumes are desirable to minimize analyst exposure. Aside from safety factors, injection volumes greater than 1 mL are not recommended due to column and detection volume capacity. Peak resolution and quantitation may be distorted by large injection volumes. To adjust the gain setting, press <GAIN> in the *Set Up* section of the Photovac top panel. Increase the gain setting to 50 by depressing the up arrow key.

   When "50" appears on the LCD, press <ENTER>. Perform a pre-operational checkout (Section 5.7.2) to fine tune carrier flow, if necessary. Otherwise, proceed with instrument settings.

5. To set the chart recorder, press <CHART>. Using arrow keys, obtain LCD readout "Chart Recorder on with Baseline." Press <ENTER>. The baseline mode is recommended because it allows the operator to observe integration parameters and make adjustments when necessary. "Speed? cm/min" appears on the LCD. Use arrow keys to obtain 0.5 cm/min on the LCD, and press <ENTER>.

6. To enter peak integration parameters, press <SENS>. When prompted by the LCD, using the arrow keys and <ENTER> adjust settings to:

   UPSLOPE: 18
   DOWNSLOPE: 14
   PW @ 4: 6

   "Upslope" and "downslope" refer to the change in baseline slope necessary for the integrator to recognize a beginning and end of a peak. The downslope is kept lower than the upslope so the tails of peaks are fully integrated. "PW@4" refers to the peak width in seconds at 4 min. This value is proportionally adjusted by the integrator for retention times other than 4 minutes.

7. The peak window settings are pertinent only if using the internal Photovac microprocessor's library. Peak identification in gas chromatography is based on retention time (RT) matches with the standards used. When operating at "ambient" temperature, fluctuations in external temperature will affect compound retention times, making peak identification difficult and questionable. It is for this reason that the Photovac should be operated in a stable temperature environment. Press <WINDO>, using the arrow keys and <ENTER> to adjust the settings for the packed column to 10 seconds and for the capillary column to 5 seconds.

   The internal microprocessor applies an equation allowing for more extensive fluctuations in RTs of later eluting compounds relative to early eluting compounds, using the window setting selected.

8. The area of rejection setting is used to eliminate the reporting of "noise" peaks on the report printed at the end of each run. It designates the minimum peak area (volt - seconds) recognized by the integration system. A setting of 100 mVs is usually sufficient for detection of aromatics and chlorinated alkenes at the 5-20 ppb level at a gain setting of 50.

   Press <AREA>. Press the arrow keys until 100 mVs appears on the LCD. Press <ENTER>.

9. Events (Manual Injection)

   - Manual injection operation of the Photovac 10S series with serial flow only involves EVENT (valve actuators) 1 (a pump, with audible sound used only for injection timing). EVENT 1, the pump timer, is set for a recommended maximum of 2 seconds.

- Press <EVENT>. When prompted by the LCD readout, make the following entries:

EVENT #	ON (Sec)	OFF (Sec)
1	5	7
2	0	0
3	0	0
4	0	0
5	0	0
6	0	0
7	0	0
8	0	0

- Pressing <STATUS/TEST>, then <ENTER> produces a hard copy of the events just entered to check for accuracy of entries and to document analytical procedures.

10. The Photovac GC analysis run time (length of time peaks are recognized for integration by the internal microprocessor) is set using the *Cycle* key. Press <CYCLE>. The LCD will prompt you for "Timer Delay (sec)." The number of seconds entered (by the numeric keys) will be the length of time between pressing the start key and the start of the chart record (i.e., the zero reference point for all peak retention times). This value should normally coincide with the point at which the sample is injected (i.e., the EVENT-1 "off" time). Type "7," and press <ENTER>.

11. "Analysis Time" will appear on the LCD readout. Using the numeric key pad, type "3000," and press <ENTER>. This can be shortened later. The longest analysis time possible with the Photovac is 3267 seconds (54 minutes).

12. "Cycle Time" appears on the LCD. Type "0" and press <ENTER>.

13. Perform a baseline check for Photovac operational readiness. To determine run time progression, press the up arrow key. For operational readiness, runs need only be 600-700 seconds. An initial negative baseline indicates the detector is still "warming up." An elevated or irregular baseline, after proper flow adjustments, indicates possible contamination which may require additional purging time. After a suitable baseline has been obtained, the instrument is ready for calibration.

5.7.5 Calibration

Refer to Table B, Appendix B, for the calibration and maintenance schedule. Photovac Incorporated conducts an instrument calibration and includes the chromatogram as a component of the instruction manual. Check the instrument's performance by duplicating the factory calibration check and comparing the results.

1. Take a clean 1-L sample bottle or a clean 1-L Tedlar bag fitted with a septum cap, and completely flush with a good quality bottled air.

2. Using the factory calibration data sheet, calculate the required amounts of each calibration compound required to generate an air standard (with a total volume of 1 liter) identical to that run by Photovac in the factory calibration. Refer to section 7.0 in the Photovac manual (titled Calculations) if a suitable gas standard mixture is not available.

3. Using an appropriate volume gas-tight syringe, aspirate the required amount of each compound from the headspace of the storage bottles at room temperature, and inject it into the purged 1-L sample bottle or Tedlar bag. Be careful to fully flush the syringe with clean air between each compound. Fill the Tedlar bag with the factory calibration standard.

4. Allow 10 minutes for the standard to equilibrate.

5. Using a clean 100-μL, gas-tight syringe, aspirate the required injection volume from the 1-L standard. With a crisp, snappy action, inject the standard into the proper "injection port" of the Photovac.

6. Compare the chromatograph generated with the factory-supplied "specification chromatogram." If the difference is significant, review the procedures and technique used in the analysis and repeat. If results are still unsatisfactory, call Photovac technical service.

Alternate Procedures for Calibration

1. Following the start up procedure in the instruction manual, get the Photovac on line ready to accept a sample.

2. Obtain a gas standard mixture certified to ±2% accuracy, commercially available from Matheson Gas Products or equivalent.

3. Using a clean, 100-μL, gas-tight syringe, aspirate the required injection volume from the standard. With a crisp, snappy action, inject the standard into the proper "injection port" of the Photovac.

4. Compare the chromatograph generated with the factory-supplied "specification chromatogram." If the difference is significant, review the procedures and technique used in the analysis and repeat. If results are still unsatisfactory, call Photovac technical service.

5.7.6 Shut Down

1. Press <OFF>, then press <ENTER>.

2. Reset the carrier gas flow to 2-5 cm^3/min.

3. Place instrument on charge while on the bench and maintain as described in Section 5.7.7.

4. Unplug the unit except when charging batteries.

5.8 CALCULATIONS

5.8.1 Calibration Curve

A calibration curve of at least three concentrations must be constructed for each target compound. A straight line equation in the form of y = (m)(x) + b, (where: x = concentration, y = area counts, m = slope and b = the intercept) is fit to the standards' raw data. The (y), or the unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative of detector response range limitations.

Alternatively, sample concentration can be calculated by:

$$[Sample] = [Std] \frac{A_1}{A_2} \cdot \frac{V_2}{V_1}$$

where:

sample = concentration of sample in ppm or ppb
A_1 = peak area of sample (volts x seconds)
A_2 = peak area of standard (volts x seconds)
V_1 = injection volume of sample (μL)
V_2 = injection volume of standard (μL)
std = concentration in ppm or ppb

5.8.2 Standard Response Generation/Duplication of Factory Calibration Data

If appropriate gas standard mixtures are not available, gas standards can be made using the headspace from 40-mL VOA bottles with Teflon-lined septa screw caps partially filled with the desired neat volatile liquid. Generally, factory instrument response is determined using the following three compounds:

Compound	P_{vap} @ 20° C
methylene chloride	347 mm Hg
n-hexane	126 mm Hg
benzene	74 mm Hg

These compounds are toxic and should be stored and worked with under a hood. The general formula for preparing a standard from the headspace above a volatile liquid is:

$$V_{HS} = \frac{760\ (C)\ (V)}{P_{vap}}$$

where:

V_{HS} = volume of headspace (μL)
P_{VAP} = vapor pressure of liquid (mm HG)*
C = desired concentration (ppm)

V = volume of standard vessel (liters)

• Use appropriate tables to determine compound vapor pressure if working environment is not 20° C.

A determined volume of neat liquid headspace may be introduced to the standard vessel through the septum if using a Tedlar bag with the appropriate fitting. Bags or vessels used should be labelled with content concentrations, date, and time of preparation.

5.9 QUALITY ASSURANCE/ QUALITY CONTROL

There are no specific quality assurance activities which apply to the operation of the Photovac. However, all instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

5.10 DATA VALIDATION

This section is not applicable to this SOP.

5.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific health and safety practices.

6.0 PHOTOVAC GC ANALYSIS FOR AIR, SOIL GAS, WATER, AND SOIL: SOP #2109

6.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes a low-cost field laboratory screening tool for tentative identification and determination of concentration levels of select contaminants for site assessment and health and safety surveys.

This method describes the rapid screening of air, soil gas, water, and soil samples using a Photovac portable gas chromatograph (GC) model 10S series to determine the presence of various volatile organic compounds.

The data allows only rapid evaluation of site conditions and is applied to, but not limited to, the following activities: determining the extent and degree of contamination; defining the pollutant plume; assessing health and safety; and tentatively identifying and quantifying pollutants. The data should not be used for site ranking or for enforcement, since only limited QA/QC is required, and the reported data is qualified as "tentative."

6.2 METHOD SUMMARY

Air, soil gas, water, or soil samples can be analyzed by the Photovac. Brief method summaries are provided below. All methods use a Photovac 10S series GC equipped with a 10.6 eV photoionization detector (PID), and use external standards to tentatively identify and quantify compounds of interest. Refer to ERT SOP #2108, Photovac 10S50, 10S55 and 10S70 Gas Chromatograph Operation for additional information.

6.2.1 Air and Soil Gas Samples

Ambient air or soil gas samples are collected in 1-L Tedlar bags. An aliquot of each bag sample is withdrawn using a gas-tight syringe and directly injected into the GC. Vapor from selected samples can then be absorbed onto Tenax/CMS cartridges for confirmational GC/MS analysis.

6.2.2 Water Samples

Water samples are collected in 40-mL VOA vials with Teflon-lined, silicone septum screw caps. A 20-mL aliquot of sample is transferred by pipette into a second, clean VOA vial. The vial is capped, shaken vigorously for one minute, and allowed to stand at room temperature for at least 30 minutes for vapor phase equilibration. An aliquot of the water headspace is then injected into the GC using a gas-tight syringe.

6.2.3 Soil Samples

Soil samples are also collected in VOA vials. A 5 g aliquot of sample is weighed into a second, clean vial. Enough reagent water is added to bring the total volume of the soil/water extract to 20 mL. The vial is then capped, shaken vigorously for 1 minute, and allowed to stand at room temperature for at least 1 hour for vapor phase equilibration. An aliquot of the soil headspace is then injected into the GC using a gas-tight syringe.

6.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

6.3.1 Air and Soil Gas Samples

Air and soil gas samples are collected and stored in 1-L Tedlar gas sampling bags, using procedures outlined in ERT SOP #2149, Soil Gas Sampling. Samples should be kept in a cooler out of direct light and heat. Samples should be analyzed within 48 hours of collection, preferably within 12 hours.

Alternatively, samples may be collected in SUMMA canisters (see ERT SOP #1704, SUMMA Canister Sampling). In this case, sample stability may extend up to 2 months, depending upon sample matrix.

6.3.2 Water Samples

Water samples are collected, in triplicate, in 40-mL VOA vials. One sample is analyzed by the Photovac; the two remaining vials are used for

confirmation analysis by another method. They are filled completely, with no visible air bubbles. Samples are immediately stored out of direct light, in a cooler packed with ice from the time of collection until analysis. Sample vials are protected against breakage, and analyzed within seven days of collection.

6.3.3 Soil Samples

Soil samples are collected in 40-mL VOA vials, and stored out of direct light, in a cooler packed with ice (see ERT SOP #2012, Soil Sampling). Sample containers need to be protected from breakage.

6.4 INTERFERENCES AND POTENTIAL PROBLEMS

6.4.1 All Samples

- High concentrations of short chain alkanes and alkenes in samples may interfere with the resolution and detector sensitivity of early-eluting chlorinated alkenes and aromatic compounds.

- Syringes may cause carryover contamination between samples. This can be monitored by running regular syringe blanks and can be minimized by decontaminating syringes properly between samples.

- Since the Photovac is a GC, the target compounds are identified by their retention times (RTs). If the RT of the sample peak(s) matches the RT of the standard peak(s), they are assumed to be identical. If any non-target compound has the same RT, it can be misidentified as a target compound.

6.4.2 Air and Soil Gas Samples

- Samples can be contaminated by diffusion of volatile organics through the septum seals and the walls of the sampling bag during shipment and storage. A field blank (clean Tedlar bag filled with ultrazero air, carried through sampling and handling protocol) can serve as a check on such contamination.

- To prevent contamination by off-gassing, use Teflon or equivalent inert fittings and tubing in all procedures.

6.4.3 Water and Soil Samples

- Liquid samples should not be directly introduced into the GC. Direct injection of liquids, without heated injection ports, may result in damage to the GC.

- Samples can be contaminated by diffusion of volatile organics through the septum seal during shipment and storage. A field reagent blank (a clean sample container filled with reagent water, carried through sampling and handling protocol) can serve as a check on such contamination.

- Some of the sample will volatilize when the vials are opened during sample preparation. This loss is minimized by proper sample handling.

6.5 EQUIPMENT/APPARATUS

6.5.1 Photovac Operation

- Photovac 10S series gas chromatograph, with power cord and manual
- extra source lamp
- Photovac lamp tuning screwdriver
- extra columns/fittings
- ultrazero air carrier gas
- two-stage regulator, with quick-connect fitting
- one flowmeter per Photovac, either bubble-meter, rotameter, or Gilibrator
- septa, 6-mm diameter
- syringes, gas-tight, 10 μL to 1 mL
- VOA vials filled with activated charcoal, for syringe cleaning
- integrator or strip-chart recorder, with appropriate connections
- extra Photovac integrator pens
- extra Photovac integrator paper
- labels
- tools -- large adjustable wrench, wrenches (5/16 inch to 9/16 inch), screwdrivers (flat and Phillips head), needle-nose pliers, jeweler's screwdrivers, Allen wrenches
- duct tape
- Teflon tape

- power strip
- snoop
- Kimwipes (or similar lint/static free wipe) and
- Pelican cases (or equivalent)

6.5.2 Soil Gas Analysis

- Tedlar bags, 1 liter
- SUMMA canisters for holding gas standards
- extra-large syringe, 100 mL to 500 mL, for serial dilutions

6.5.3 Tenax/CMS Sampling

- Tenax/CMS cartridges in sealed glass ampules
- culture tubes (labeled) with glass wool to ship cartridges
- cotton gloves or cloths for cartridge handling
- fitting to connect syringe to cartridge
- fitting to connect cartridge to Tedlar bag
- 1/4-inch silicone o-rings for a tight seal around cartridge

6.5.4 Water Headspace Analysis

- headspace standards, purgeable A and B or equivalent
- 1.8-mL vials for holding standards (either screw-cap or crimp-top vials)
- Pasteur pipettes for transferring standards
- 40-mL VOA vials (1 per sample plus extras for standards and QA/QC requirements)
- 10-mL or 20-mL pipettes and pipette bulb
- liquid standard syringes
- surgical gloves

6.5.5 Soil Headspace Analysis

- same equipment for water headspace analysis
- portable scale, accurate to ± 0.1 g
- spatulas, or equivalent, for transferring soil

6.6 REAGENTS

6.6.1 Air Sample Analysis

- gas standards -- certified to ± 2% level of accuracy. Although there may be other sources, gas standards are available through Scott Specialty Gas. In-house laboratory preparation of calibration gas standards with confirmational GC/MS analysis is acceptable

- ultrazero air carrier gas

6.6.2 Water and Soil Sample Analysis

- reagent water -- organic-free chromatographic grade or equivalent, free of any contaminants which may interfere with the detection and resolution of target parameters

- ultrazero air carrier gas

- stock standard solutions -- stock standard solutions may be prepared from pure standard materials or purchased as certified solutions (e.g., Supelco purgeable A or B, or equivalent). Reagents used as standards may depend on site-specific suspected volatile contaminants

6.7 PROCEDURES

6.7.1 Method Detection Limits

Determine the method detection limit (MDL) just before the analysis with a serial dilution of the standard. The MDL is the lowest concentration that can be detected at the gain setting selected for the analysis. MDLs depend on the type of analysis performed and the condition of the gas chromatograph. Because of the difference in matrices, air and soil gas analyses usually have MDLs an order of magnitude above headspace analyses. Factors that can vary the sensitivity of a Photovac from site to site are the age of the source lamp, detector age, column condition, shipment of GC to the site, and location of the field lab. Typical MDLs for soil gas range from 10 ppb to 50 ppb, and headspace MDLs range from 1 ppb to 5 ppb.

If sample concentrations are high, injection volumes may be reduced to obtain on-scale response for parameters of interest, and to avoid contamination of the GC system. Calculate the method detection limit for compounds not detected at reduced injection volumes by using:

$$MDL = \frac{(V_{std})\ (C_{std})}{V}$$

where:

V_{std} = lowest volume of standard headspace injected (μL)
C_{std} = concentration of standard (ppm or ppb)
V = volume of sample headspace injected (μL).

6.7.2 Calibration

Photovac analyses are calibrated by the external standard method using the gas standards described in Section 6.6.1. At the beginning of the analysis, a three-to-five point calibration curve is run to demonstrate linear instrument response over a specified concentration range. The development of this method has shown the best linearity of the PID response to be from 10 ppb to 1 ppm for air and soil gas analysis and from 1 ppb to 100 ppb for headspace analysis. Most PIDs will be linear above that range but eventually, at high enough concentrations, the PID will become saturated. The curve is verified daily by running a calibration check standard from the middle of the curve.

If the response of any parameter varies from the curve by more than ±25%, RSD instrument response has changed and a new calibration curve should be run.

Air and Soil Gas Calibration

Prepare the concentrations needed for calibration by performing a serial dilution of the gas standard. For example, add 50 mL of a 1-ppm standard and 450 mL of ultrazero air carrier gas to a new Tedlar bag to acquire a 100-ppb calibration standard. The 100-ppb bag can then be used to make up lower concentration standards.

Alternatively, construct a calibration curve by varying injection volumes. By designating a 250-μL injection volume as the 1-ppm standard, a 100-ppb standard is created by injecting 25 μL of a 1-ppm standard. This method is more convenient and does not require the large syringes needed for serial dilution, but the calibration curve is then limited by the sizes of the available syringes.

Water and Soil Calibration

Headspace standards can be created at selected concentrations by adding the appropriate volumes of stock standard into clean 40-mL VOA vials containing 20 mL of reagent water. These volumes (V) are calculated by using:

$$V = \frac{20\ mL \cdot (stock\ conc.)}{(calibrant\ conc.)}$$

From the 200-ppm purgeable A and B standards, first prepare a 2-ppm stock solution to allow calibration standards between 1 ppb and 10 ppb to be prepared with the syringes listed in Section 6.5.1.

6.7.3 Operation

Air and Soil Gas Analysis

Typical columns used for this method include SE-30 (packed) and CP-Sil 19 (capillary). An example of compound separation using CP-Sil 19, with typical chromatographic conditions, is shown in Figure 1, Appendix A.

1. Inject standards after every 10-15 samples or every 6 hours, whichever is more frequent, to bracket possible parameter RT variations.

2. If sample concentrations are high, reduce injection volumes to obtain on-scale response. The method detection limit (MDL) for compounds not detected at reduced injection volumes is calculated according to the equation in Section 6.7.1.

3. Identify the compounds in the sample by comparing the retention time of the peaks in the sample chromatogram with those of the peaks in the standard chromatograms. The width of the RT windows used to make identifications should be based on measurements of actual RT variations of standards which bracket a series of sample injections. Three times the standard deviation of a retention time can be used to calculate a suggested window size; however, the judgment of the analyst should be a major factor in the interpretation of chromatograms.

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COMPENDIUM OF ERT AIR SAMPLING PROCEDURES

SUMMA Canister Cleaning

SUMMA Canister Sampling

GC/MS Analysis of Tenax/CMS Cartridges and SUMMA Canisters

Preparation of SUMMA Canister Field Standards

Low Level Methane Analysis for SUMMA Canister Gas Samples

Asbestos Sampling

Tedlar Bag Sampling

Charcoal Tube Sampling

Tenax Tube Sampling

Polyurethane Foam Sampling

Interim Final

Environmental Response Team
Emergency Response Division

Office of Emergency and Remedial Response
U.S. Environmental Protection Agency
Washington, DC 20460

Notice

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Each Standard Operating Procedure in this compendium contains a discussion on quality assurance/quality control (QA/QC). For more information on QA/QC objectives and requirements, refer to the *Quality Assurance/Quality Control Guidance for Removal Activities*, OSWER directive 9360.4-01, EPA/540/G-90/004.

Questions, comments, and recommendations are welcomed regarding the Compendium of ERT Air Sampling Procedures. Send remarks to:

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Acknowledgments

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1.0 SUMMA CANISTER CLEANING: SOP #1703

1.1 SCOPE AND APPLICATION

This procedure is intended for use when cleaning SUMMA polished stainless steel canisters. SUMMA canisters are able to sample gas-phase volatile organic compounds (VOCs) on site at concentrations of one part per billion by volume (ppbv) and greater. This cleaning procedure ensures that canisters have been sufficiently cleaned prior to sampling, to the extent that no VOC contamination is present at concentrations greater than 0.2 ppbv.

1.2 METHOD SUMMARY

After use, canisters are logged in and physically inspected. These canisters are vented to the outside air under an operating exhaust hood. Canisters are connected to a manifold which is attached to a vacuum pump via a cryogenic trap. The canisters and lines are evacuated and then the canisters are heated for a prescribed time period. During the heating period, the canisters are filled with humidified nitrogen and pressurized. Three cycles of filling and pressurizing, then evacuation and heating, are required.

Confirming that the canisters are free of VOC contamination involves pressurizing the canisters with ultrahigh purity nitrogen and analyzing on the gas chromatograph/mass spectrometer (GC/MS). If no VOC contamination is present at concentrations greater than 0.2 ppbv, the canister is considered clean. Clean canisters are leak-tested by pressurizing with nitrogen for 24 hours. Canisters that have been cleaned and found to be without leaks are evacuated. These canisters are logged as cleaned and certified and are stored in the evacuated state with brass cap fittings until needed for sampling.

1.3 SAMPLE CANISTER HANDLING AND STORAGE

1.3.1 Canister Receipt

1. Observe the overall condition of each sample canister. Any canister having physical defects requires corrective action.

2. Observe each canister for an attached sample identification number.

3. Record each canister in the dedicated laboratory logbook by its SUMMA canister number.

1.3.2 Canister Storage

1. Store canisters in an evacuated state of less than 0.05 mm Hg and with a brass cap in place. The canisters remain in this state until needed.

2. Attach an identification tag to the neck of each canister for field notes and chain-of-custody purposes.

3. Record each canister in the dedicated laboratory logbook stating the canister status and storage location. Also, note on the identification tag the date cleaned and date certified clean, as well as the initials of the operator.

1.4 INTERFERENCES AND POTENTIAL PROBLEMS

Contamination may occur in the sample canisters if they are not properly cleaned before use. All other equipment used in this process must be sufficiently clean. All gases and solvents used must be of a certified purity to avoid contamination. Canisters must be stored with the valve closed and the brass caps in place to avoid vacuum loss.

1.5 EQUIPMENT/APPARATUS

1.5.1 Canister

- sample canister -- leak-free stainless steel pressure vessels of desired volume (e.g., 6-L), with valve and SUMMA passivated interior surfaces or equivalent.

Although there maybe other sources, two readily available sources are Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID, 83843; or Andersen Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA, 30315.

1.5.2 Canister Cleaning System

Figure 1 in Appendix A displays the canister cleaning system.

- vacuum pump -- capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.
- manifold -- stainless steel manifold with connections for simultaneously cleaning several canisters.
- shutoff valve(s) -- three on/off toggle valves (Valves A, B, D).
- shutoff valve -- one variable metering valve (Valve C) to regulate flow of zero air.
- shutoff valve -- one variable metering valve (Valve E) used as an on/off valve between the nitrogen regulator and the supply line.
- stainless steel vacuum gauge -- capable of measuring vacuum in the manifold to an absolute pressure of 0.05 mm Hg or less.
- cryogenic trap -- stainless steel U-shaped open tubular trap cooled with liquid nitrogen to prevent contamination from back diffusion of oil from vacuum pump. Also, a stainless steel two-stage pressure regulator 0-690 kPa (0-100 psig) to regulate nitrogen pressure.
- Teflon tee with a septum port -- an injection port capable of introducing distilled, deionized water to provide moisture to the zero air supply line.
- isothermal oven -- a system for canisters or equivalent. Although there may be other sources, one readily available source is Fisher Scientific, Pittsburgh, PA, Model 349.

1.6 REAGENTS

- gas cylinders of nitrogen, ultrahigh purity grade.
- cylinders of liquid nitrogen, ultrahigh purity grade.
- cryogen -- liquid nitrogen (bp -195°C).
- distilled, deionized water, ultrahigh purity.

1.7 PROCEDURES

1.7.1 System Set-Up

1. Seal all connections in the vacuum system except the canisters and manifold. Check all connections, lines, and valves for leaks by pressurizing the line to 30 psig and using a soap solution. Check the septum for leaks by removing it and visually inspecting it.
2. Add the liquid nitrogen to the cryogenic trap and allow it to reach a state of equilibrium.
3. Check the pump to assure proper working order by achieving a vacuum of 0.05 mm Hg in the line that normally attaches to the manifold but is now capped. Valve A is open and Valves B, C, D, and E are closed. After the vacuum test is completed, turn the pump off and remove the cap to break the vacuum.
4. Check the oven to assure proper working order by heating the oven to 100°C and measuring the internal temperature with a thermometer.
5. Check reagents to assure proper purity.
6. Set the back pressure on the nitrogen to 30 psig.

1.7.2 Cleaning

1. Vent all canisters to the outside air under an operating exhaust hood.
2. Connect the canisters (with the valves closed on the canisters) to the manifold by the Swagelok fittings. Connect the manifold to the vacuum system by the Swagelok fitting.

3. Open Valve A, ensure Valves B, C, D, and E are closed, and start vacuum pump.

4. Once a vacuum (0.05 mm Hg) is obtained in the line and the manifold, close valve A. Examine the system for leaks by comparing the initial vacuum reading and a second vacuum reading 3 minutes later. If the vacuum deteriorates more than 5 mm Hg, a leak exists and corrective action is necessary.

5. If no leaks are observed, open valve A and the Canister 1 valve. Evacuate Canister 1 to 0.05 mm Hg, then close the Canister 1 valve. By evacuating one canister at a time, the potential for cross-contamination between canisters is minimized.

6. Evacuate all other canisters in the same manner as described in step 5.

7. After all four canisters are evacuated, open all canister valves. Turn on the oven and heat to 100°C.

8. Continue evacuating canisters for 1 hour at 100°C. Document the time.

9. After 1 hour, Valve A is closed and Valves B, C, D, and E are opened, with Valve C metering the flow of nitrogen.

10. Inject 400 µL of distilled deionized water via a syringe through the septum in the nitrogen line.

11. Allow the canisters to pressurize to 30 psig.

12. Close Valves B, C, D, and E.

13. Close canister valves.

14. Repeat steps 5 through 13, twice.

15. Close valves on canisters.

16. Close Valve A.

17. Turn off vacuum pump.

18. Disconnect manifold from cleaning system.

19. Disconnect canisters from the manifold and place a brass cap on each canister.

20. Choose one canister of this set of four that was analyzed as being the most highly contaminated previous to cleaning. Fill this canister with ultrahigh purity nitrogen air to a pressure of 30 psig.

21. Analyze the above canister for VOC contamination by GC/MS. If this canister is sufficiently clean to the extent that no VOC contamination is present at concentrations greater than 0.2 ppbv, then all canisters in that set of four are considered clean. Document the results. If it is not sufficiently clean, see step 23.

22. Evacuate the above canister again to 0.05 mm Hg, cap it with a brass fitting, and store it with the other three of the lot. Document the location.

23. If the above canister is not sufficiently clean (i.e., VOC contamination is present at concentrations greater than 0.2 ppbv), then all canisters in that lot must be cleaned again until the canisters meet the prescribed criteria. Document the results.

1.7.3 Leak-Testing

1. Once the canister lot is determined to be clean, the canisters are pressurized to 30 psig with nitrogen.

2. The initial pressure is measured, the canister valve is closed, and the brass cap is replaced. Document the time and pressure.

3. After 24 hours, the final pressure is checked. Document the time and pressure.

4. If leak-proof, the pressure should not vary more than +13.8 kPa (± 2 psig) over the 24-hour period. If this criterion is met, the canister is capped with a brass fitting and stored. If a leak is present, corrective action is required. Document the results.

1.8 CALCULATIONS

There are no calculations for this SOP.

1.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following specific quality assurance/quality control procedures are applicable for SUMMA canister cleaning:

1. Check all connections, lines, and valves to ensure no leaks are present.

2. Check the septum to ensure no leaks are present, by removing the septum and visually examining it.

3. Check the pump to ensure proper working order by achieving a vacuum of 0.05 mm Hg prior to cleaning.

4. Check the oven to ensure proper working order by comparing the oven setting at 100°C to the internal temperature with a thermometer.

5. Check the reagents to ensure sufficient purity.

6. Evacuate all canisters to 0.05 mm Hg during each cycle of the cleaning process and document the results.

7. Evacuate all canisters at 100°C for 1 hour during each cycle of the cleaning process. Document the results.

8. Evacuate, heat, and pressurize all canisters three times during the cleaning process. Document each cycle.

9. For the canister lot to be considered cleaned, the selected canister from the cleaning lot to be tested must be analyzed by GC/MS and shown to be sufficiently cleaned to the extent that no VOC contamination is present at concentrations greater than 0.2 ppbv. If the VOC contamination is greater than 0.2 ppbv, the canister lot must be cleaned again. In either case, document the results.

10. Leak-test all canisters for 24 hours and document the results.

11. Store and evacuate all canisters, and cap them with a brass fitting. Document the pressure and location of all canisters.

1.10 DATA VALIDATION

This section is not applicable to this SOP.

1.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific safety practices. More specifically:

- Liquid nitrogen is used to cool the cryogenic trap. Its boiling point is -196°C. Insulated gloves, lab coat, face shield, and safety glasses must be worn when using this material. Liquid nitrogen must be transported only in properly constructed containers.

- Ultrahigh purity nitrogen is used to clean the canisters and must be labeled properly. All cylinders must be securely fastened to a stationary object. The cylinder valve should only be opened by hand. The proper regulator must be used and set correctly.

- The oven is set to a temperature of 100°C. Insulated gloves should be worn when handling items heated to this temperature.

- Prior to cleaning, canisters are to be vented to the atmosphere under an operating exhaust hood. The hood must be in proper working order.

- Canisters are pressurized during the cleaning operation. No canister is to be pressurized above 30 psig. The maximum pressure limit for the SUMMA canisters is 40 psig.

2.0 SUMMA CANISTER SAMPLING: SOP #1704

2.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe a procedure for sampling of volatile organic compounds (VOCs) in ambient air. The samples are collected as whole air samples in passivated SUMMA stainless steel canisters. The VOCs are subsequently separated by gas chromatography (GC) and measured by mass-selective detector or multidetector techniques. This SOP describes procedures for sampling with canisters at final pressures both above atmospheric pressure (referred to as pressurized sampling) and below atmospheric pressure (referred to as subatmospheric pressure sampling).

This method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. The organic compounds that have been successfully collected in pressurized canisters by this method are listed in table 1, Volatile Organic Compound Data. These compounds have been measured at the parts per billion by volume (ppbv) level.

2.2 METHOD SUMMARY

Both pressurized and subatmospheric pressure sampling modes use an initially evacuated canister. Both modes may also use a mass flow controller/sample pump arrangement, fixed orifice, capillary, or adjustable micrometering valve to regulate flow. With this configuration, a sample of ambient air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated passivated SUMMA canister.

2.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

After the air sample is collected, the canister's valve is closed, an identification tag is attached to the canister, and the canister is transported to a laboratory for analysis. Upon receipt at the laboratory, the canister tag data are recorded. Sample holding and expiration times should be determined prior to initiating field activities.

2.4 INTERFERENCES AND POTENTIAL PROBLEMS

Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned. Instructions for cleaning the SUMMA canisters are described in ERT SOP #1703, SUMMA Canister Cleaning.

2.5 EQUIPMENT/APPARATUS

See figure 2 for a diagram of pressurized and subatmospheric canister sampling systems.

2.5.1 Subatmospheric Pressure Sampling Equipment

- VOC canister sampler -- whole air sampler capable of filling an initially evacuated canister by action of the flow control from near 30 inches of mercury (Hg) vacuum to near atmospheric pressure (such as Andersen Samplers, Inc., NuTech, Scientific Instrumentation Specialists (SIS), or homemade subatmospheric canister samplers).

- sampling inlet line -- stainless steel tubing to connect the sampler to the sample inlet.

- sample canister (6-liter size) -- leak-free stainless steel pressure vessels of desired volume with valve and SUMMA passivated interior surfaces (SIS, Andersen Samplers, Inc., or equivalent).

- particulate matter filter -- 2-μm sintered stainless steel in-line filter (Nupro Co., Model SS-2F-K4-2, or equivalent).

- chromatographic-grade stainless steel

Table 1: Volatile Organic Compound Data Sheet

Compound Name (synonym)	Formula	Molecular Weight	Boiling Point (°C)	Melting Point (°C)	CAS Number
Freon 12 (dichlorodifluoromethane)	Cl_2CF_2	120.91	-29.8	-158.0	------
methyl chloride (chloromethane)	CH_3Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	-94.0	------
vinyl chloride (chloroethylene)	$CH_2=CHCl$	62.50	-13.4	-1538.0	75-01-4
methyl bromide (bromomethane)	CH_3Br	94.94	3.6	-93.6	74-83-9
ethyl chloride (chloroethane)	CH_3CH_2Cl	64.52	12.3	-136.4	75-00-3
Freon 11 (trichlorofluoromethane)	CCl_3F	137.38	23.7	-111.0	------
vinylidene chloride (1,1-dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	-122.5	75-35-4
dichloromethane (methylene chloride)	CH_2Cl_2	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	47.7	-36.4	------
1,1-dichloroethane (ethylidene chloride)	CH_3CHCl_2	98.96	57.3	-97.0	74-34-3
cis-1,2-dichloroethylene	$CHCl=CHCl$	96.94	60.3	-80.5	------
chloroform (trichloromethane)	$CHCl_3$	119.38	61.7	-63.5	67-66-3
1,2-dichloroethane (ethylene dichloride)	$ClCH_2CH_2Cl$	98.96	83.5	-35.3	107-06-2
methyl chloroform (1,1,1-trichloroethane)	CH_3CCl_3	133.41	74.1	-30.4	71-55-6
benzene (cyclohexatriene)	C_6H_6	78.12	80.1	5.5	71-43-2
carbon tetrachloride (tetrachloromethane)	CCl_4	153.82	76.5	-23.0	56-23-5
1,2-dichloropropane (propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.4	-100.4	78-87-5
trichloroethylene (trichloroethene)	$ClCH=CCl_2$	131.29	87.0	-73.0	79-01-6
cis-1,3-dichloropropene (cis-1,3-dichloropropylene)	$ClCH_2CH=CHCl$	110.97	76.0	-----	------

tubing and fittings for interconnections -- all materials in contact with sample, analyte, and support gases should be chromatographic-grade stainless steel.

- fixed orifice, capillary, or adjustable micrometering valve -- used in lieu of the electronic flow controller/sample pump for grab samples or short duration time-integrated samples.

2.5.2 Pressurized Sampling Equipment

- VOC canister sampler -- whole air sampler capable of filling an initially evacuated canister by action of the flow controller and pump from near 30 inches Hg vacuum to 15-20 psi atmospheric pressure (Andersen Samplers Inc., NuTech, SIS, or equivalent pressurized canister sampling system).

- mass flowmeter/controller -- leak-free, linearly proportioned mass flowmeter/ controller unit at desired flowrate (e.g., 100 mL/min). Although there may be other sources, a mass flowmeter/controller is available from Tylan, 15 Meadowview Ln, Medford, NJ 08055.

- sampling inlet line -- stainless steel tubing to connect the sampler to the sample inlet.

- sample canister -- leak-free stainless steel pressure vessels of desired volume with valve and SUMMA passivated interior surfaces (SIS, Andersen Samplers, Inc., or equivalent).

- particulate matter filter -- 2-μm sintered stainless steel in-line filter (Nupro Co., Model SS-2F-K4-2, or equivalent).

- chromatographic-grade stainless steel tubing and fittings for interconnections -- all materials in contact with sample, analyte, and support gases should be chromatographic-grade stainless steel.

2.6 REAGENTS

This section is not applicable to this SOP.

2.7 PROCEDURES

2.7.1 Subatmospheric Pressure Sampling

1. Prior to sample collection, complete the appropriate information on the Canister Sampling Field Data Sheet (Appendix C).

2. Open a canister, which is evacuated to 28-30 inches Hg at sea level and fitted with a flow restricting device, to the atmosphere containing the VOCs to be sampled. The pressure differential causes the sample to flow into the canister. (Note: at higher elevations the vacuum may be less.) See section 2.8 to calculate the flow rate.

3. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours). Sampling duration depends on the degree to which the flow is restricted. The flow will remain constant until the vacuum reads approximately 11 inches Hg. When this occurs, control the flow, either manually or automatically, to achieve constant flow.

4. After sampling is complete, record the appropriate information on the Canister Sampling Field Data Sheet.

2.7.2 Pressurized Sampling

1. Prior to sample collection, complete the appropriate information on the Canister Sampling Field Data Sheet.

2. Use a digital time-programmer to pre-select sample duration, and start and stop times.

3. Open a canister, which is evacuated to 28-30 inches Hg at sea level and connected in line with the sampler, to the atmosphere containing the VOCs to be sampled.

4. Using a direct drive blower motor assembly, draw a whole air sample into the system through a stainless steel inlet tube. (Some units do not have a blower.)

5. Using a specially modified inert sample pump in conjunction with a flow controller, pull a small portion of this whole air sample from the

inlet tube. The initially evacuated canister is filled by action of the flow controlled pump to a positive pressure not to exceed 25 psig.

6. Upon sampling completion at the location, complete the requisite information on the Canister Sampling Field Data Sheet.

2.8 CALCULATIONS

A flow control device maintains a constant flow into the canister over the desired sample period. This flow rate is determined so that the canister is filled over the desired sampling period, to 2-5 inches Hg vacuum for subatmospheric pressure sampling or to about one atmosphere (15 psi) above ambient pressure for pressurized sampling.

1. For subatmospheric sampling, the volume of the sample must be calculated before the flow rate can be determined. The sample volume can be calculated by:

$$S = V - \left(\frac{V \cdot E}{I}\right)$$

where:

S = sample volume (cm^3)
V = volume of the canister (cm^3)
I = initial canister vacuum (in. Hg)
E = estimated final vacuum (in. Hg)

For example, to calculate the sample volume of a 6-L canister with an initial canister vacuum of 28 inches Hg and an estimated final vacuum of 5 inches Hg.

$$S = 6000 - \left(\frac{6000 \cdot 5}{28}\right)$$

S = 4929 cm^3

The flow rate can be calculated by:

$$F = \frac{S}{T\ (60)}$$

where:

F = flow rate (cm^3/min or mL/min)
S = sample volume (cm^3)
T = sample period (hours)

Using a 24-hour sampling period for the above sample volume, the flow rate can be calculated as:

$$F = \frac{4929}{24 \cdot 60}$$

F = 3.42 cm^3/min

2. For pressurized sampling, only the flow rate has to be calculated.

For example, if a 6-L canister is to be filled with 12-L of sample at 2 atmospheres absolute pressure (near 30 psia) in 24 hours, the flow rate can be calculated by:

$$F = \frac{12000}{24 \cdot 60}$$

F = 8.3 cm^3/min

3. If the canister pressure is increased for analysis, a dilution factor (DF) is calculated and recorded on the sampling data sheet.

$$DF = \frac{P_f}{P_i}$$

where:

P_f = canister pressure (psig) after pressurization,
P_i = canister pressure (psig) before pressurization

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration in the sampled air.

2.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following general quality assurance procedures apply:

- All data must be documented on standard chain-of-custody forms, field data sheets, or within site logbooks.

- All instrumentation must be operated in accordance with operating instructions as

supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

2.10 DATA VALIDATION

This section is not applicable to this SOP.

2.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific health and safety practices. More specifically, pressurizing of SUMMA canisters should be performed in a well-ventilated room, or preferably under a fume hood. Care must be taken not to exceed 40 psig in the canisters. Canisters are under pressure, albeit only 20-30 psig, and should not be dented or punctured. They should be stored in a cool, dry place and always be placed in their plastic shipping boxes during transport and storage.

3.0 GC/MS ANALYSIS OF TENAX/CMS CARTRIDGES AND SUMMA CANISTERS: SOP #1705

3.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the analysis of air samples collected on either Tenax/Carbonized Molecular Sieve (CMS) cartridges or in SUMMA canisters by Gas Chromatography/Mass Spectrometry (GC/MS). These methods are applicable to volatile organic compounds (VOCs) that can be sampled by one or both of these media. The VOCs that can be routinely analyzed at the parts per billion (ppb) level for both sample collection methods are listed in table 2.

3.2 METHOD SUMMARY

These methods involve thermal desorption of cartridges or canisters into a cryogenic trap. The trap cryofocuses the sample onto the head of the analytical column, then flash heats the sample and separates it by gas chromatography. Following separation, compounds are analyzed by a positive-ion, electron-impact, mass spectrometer.

3.2.1 Tenax/CMS Cartridges

Analysis of Tenax/CMS cartridges for toxic organics in ambient air combines methods TO1 and TO2. The cartridges contain two different sorbent media. The gas sample is drawn through a glass tube containing Tenax (a porous polymer of 2,6-diphenyl phenylene oxide, the sorbent media for TO1) and Carbonized Molecular Sieve (CMS, the sorbent media for TO2). Further information on Tenax/CMS tube sampling may be found in ERT SOP #2052, Tenax Tube Sampling.

3.2.2 SUMMA Canisters

Alternatively, air samples can be collected in passivated, 6-liter, stainless steel SUMMA canisters and analyzed according to method TO14, a procedure similar to the Tenax/CMS cartridges. Information on SUMMA canister sampling may be found in ERT SOP #1704, SUMMA Canister Sampling.

3.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.3.1 Tenax/CMS Cartridges

Samples collected on Tenax/CMS cartridges are placed in clean culture tubes and forwarded as soon as possible to the laboratory. The culture tubes should be labeled and sealed with Teflon tape around the cap. Samples must be accompanied by a chain-of-custody (COC) record indicating sampling locations, sample numbers, date collected, sample matrix, and sample volumes. The COC should agree with the information on the culture tube labels, and discrepancies must be noted on the COC at the time of receipt by the laboratory. In addition, any looseness of culture tube caps or any obvious physical damage or contamination (e.g., broken cartridges, condensate in the culture tubes, or discoloration of the Tenax bed), must also be recorded on the COC.

Once samples have arrived at the laboratory, they should be refrigerated until they are analyzed. Analysis of Tenax/CMS samples must be completed within the 14-day holding time specified by TO1 and TO2. The holding time begins when the sample is first drawn onto the tube (not when the sample is received by the laboratory).

3.3.2 SUMMA Canisters

Samples collected in canisters should arrive at the laboratory with the canister valve closed and the sampling port capped. An identification tag should be attached and should agree with the information on the COC.

One of the advantages of canister samples is that they do not need any refrigeration or special handling until they are analyzed. Method TO14 does not specify a holding time for canister samples.

Table 2: Compounds Analyzed in Tenax/CMS Cartridges or SUMMA Canisters

• acetone	• chloromethane	• C_{10} terpenes
• C_2-C_8 alcohols	• chlorotoluene	• 1,1,2,2-tetrachloroethane
• C_4-C_{12} alkanes	• C_5-C_{12} cycloalkanes	• tetrachloroethene (PCE)
• C_4-C_{12} alkenes	• dibromomethane	• toluene
• C_3-C_6 alkylbenzenes	• 1,1-dichloroethane	• trans-1,2-dichloroethene
• benzene	• 1,2-dichloroethane	• 1,1,1-trichloroethane
• bromochloromethane	• C_4-C_{12} dienes	• 1,1,2-trichloroethane
• bromodichloromethane	• ethylbenzene	• trichloroethene (TCE)
• p-bromofluorobenzene	• 4-methyl-2-pentanone (MIBK)	• trichlorofluoromethane
• 2-butanone (MEK)		• trichloromethane
• carbon tetrachloride	• methylene chloride	• vinyl chloride
• chlorobenzene	• napthalene	• xylenes
• chloroethane	• styrene	

3.4 INTERFERENCES AND POTENTIAL PROBLEMS

- Structural isomers having coeluting retention times and identical mass spectra will interfere with this method. The most common interference seen in these methods is between meta-xylene and para-xylene.

- Excessive moisture in Tenax/CMS samples will cause the cryotrap to freeze, restricting sample flow from the desorber oven and resulting in poor recoveries. In general, trapping efficiencies for components with boiling points greater than water are more adversely affected than those with lower boiling points. If excessive moisture is suspected, the CMS section of the cartridge should be removed prior to sample desorption. If this step is taken, the lower boiling point compounds trapped by the CMS, such as chloromethane and vinyl chloride, will not be seen in the analysis.

- Canister samples suspected of having high concentrations of carbon dioxide (such as those collected from landfills or fire plumes), cannot be directly analyzed since the carbon dioxide will collect and freeze the cryotrap. This can be avoided by adsorbing the sample on a Tenax/CMS cartridge, which does not adsorb carbon dioxide, but retains the organic contaminants.

3.5 EQUIPMENT/APPARATUS

- GC/MS -- gas chromatograph capable of sub-ambient temperature programming interfaced with a mass spectrometric detector (Hewlett Packard 5996 GC/MS equipped with Series 1000E computer and RTE-6 software, or equivalent).

- thermal desorber -- capable of a -170°C to 250°C temperature range, equipped with GC interface (Tekmar 5010 GT automatic

thermal desorption/cryofocusing unit, or equivalent).

- chromatographic column -- capillary column, 30 m x 0.32 mm, 0.25 μm film thickness, (J & W Scientific, Inc. DB-624, or Restek, Inc. RTx-5, or equivalent).

- pre-column -- capillary fused silica column, 0.5 m x 0.32 mm, with column connector (Restek, Inc., or equivalent).

- Tenax/CMS cartridges -- 150 mg Tenax 35/50 mesh and 150 mg CMS packed into 6 x 120 mm borosilicate glass tubing with Pyrex glass wool on each end and between each phase, provided in sealed glass ampoules (Supelco, Inc., or equivalent). See the EMSL SOP for Preparation of Clean Tenax Cartridges.

- canisters -- passivated 6-liter SUMMA canisters (Andersen Samplers, Inc., or equivalent).

- mass flow controller -- 0-100 mL/min, to maintain constant flow for measuring canister sample volumes (Unit Instruments, Inc., UFC-1100 with URS 100 Readout Power Supply, or equivalent).

- stainless steel vacuum/pressure gauge -- capable of measuring 0 to 50 psi (Pennwalt Corp., Wallace and Tiernan Division, Model series 1500 dial instrument, or equivalent).

- chromatographic-grade, stainless steel tubing and stainless steel plumbing fittings.

- stainless steel cylinder regulators (5) -- two-stage pressure regulators for cylinders of helium, zero air, calibration standards, and surrogate standards.

- syringes -- 2.5-10 mL, for injecting calibration and surrogate standards (Dynatech - Precision Sampling, Inc., or equivalent).

- 9.5 mm septa (Supelco, Inc. Microsep F-174, or equivalent).

- culture tubes, Pyrex and Teflon tape -- for preserving Tenax/CMS samples.

- rotameter -- 0-100 mL/min (Matheson Gas Products, Inc., or equivalent).

- cotton cloths -- 9 inch by 9 inch, for Tenax/CMS cartridge handling (Texwipe, Co., or equivalent).

- tweezers -- for inserting and removing cartridge samples from thermal desorber.

- O-rings -- Viton, 6 mm I.D., for retaining Tenax/CMS cartridges in thermal desorber (Hewlett-Packard part no. 5061-5867, or equivalent).

3.6 REAGENTS

- calibration standards -- at approximately 1 ppmv with the balance as nitrogen (Matheson Gas Products, Inc., or equivalent).

- bromochloromethane (BCM) and p-bromofluorobenzene (BFB) -- at approximately 1 ppmv in nitrogen in a separate cylinder; both compounds used as surrogate standards, BFB also used for tuning GC/MS (Scott Specialty Gases, Inc. or equivalent).

- perfluorotributylamine (PFTBA) -- for tuning the mass spectrometer (Hewlett Packard, Inc., or equivalent).

- liquid nitrogen -- for cryogenic cooling (SOS Gases, Inc., or equivalent).

- helium -- ultrahigh purity, used as carrier gas and as purge gas in the thermal desorber (Matheson Gas Products, Inc., or equivalent).

- carbon dioxide -- bone-dry, high-pressure liquid, for chromatograph oven cooling (Matheson Gas Products, Inc., or equivalent).

- compressed air -- ultrazero grade, for chromatograph oven door control (Matheson Gas Products, Inc., or equivalent).

- nitrogen -- ultrahigh purity, for pressurizing

canister samples and purging canister analysis train lines (Matheson Gas Products, Inc., or equivalent).

3.7 PROCEDURES

3.7.1 Daily GC/MS Tuning

At the beginning of each day, tune the GC/MS system to verify that acceptable performance criteria can be achieved. The mass spectrometer should first be automatically or manually tuned on perfluorotributylamine (PFTBA). PFTBA tuning is done to demonstrate that the instrument is operating properly and, upon analysis of p-bromofluorobenzene (BFB), will give a spectrum that meets the ion abundance criteria listed in EPA Method 624 (table 3).

Table 3: GC/MS Performance Criteria for p-Bromofluorobenzene (EPA Method 624)

m/z	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	Base peak, 100% relative abundance
96	5% to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5% to 9% of mass 174
176	95% - 101% of mass 174
177	5% to 9% of mass 176

After PFTBA tuning, BFB is analyzed to check GC column performance and is used as the GC/MS performance standard. This performance test must be passed before any samples, standards, or blanks are analyzed, and must be repeated for every twelve hours of continuous operation. A background correction mass spectrum from the performance test must satisfy the criteria set forth in U.S. EPA Method 624. If the criteria are not met, the analyst must re-tune the mass spectrometer and repeat the test until all criteria are met.

3.7.2 GC/MS Calibration

1. Initial Calibration -- Before any analysis, initially calibrate the GC/MS using standards contained in pressurized cylinders at approximately 1 ppmv in nitrogen. A list of the target compounds in the calibration standards is given in table 4, along with the ions used for quantitation. A multipoint calibration is created by injecting three to five different volumes into the thermal desorber and analyzing them in the GC/MS. Typical volumes range from 1-10 mL, corresponding to concentrations of 100 ppb to 1 ppm. Following analysis of all calibration points, a calibration report is prepared listing the average response factors and their Relative Standard Deviation (RSD), which must be less than 25% for each compound. For each compound in the calibration, the retention times and relative abundances of selected ions are stored on the hard disk of the GC/MS computer to be used for compound identification.

2. Continuing Calibration -- For each day of analysis, check the GC/MS calibration before sample analysis with a daily standard, usually at the 1-ppmv concentration. The continuing calibration is only acceptable when all compound abundances in the daily standard are ± 25% of the average response factor of the calibration curve.

3.7.3 Analysis Conditions

All samples are prepared for GC/MS analysis by using a thermal desorption/cryogenic trapping unit. The unit is equipped with a 0.25-inch by 7-inch oven chamber for desorbing samples, an internal cryogenic trap (C-1) consisting of a 0.125-inch stainless-steel tube filled with Pyrex glass beads, an eight port switching valve, and an external cryogenic trap (C-2) located just above the head of the pre-column (figure 3, appendix A). A 60-inch silcosteel transfer line connects the two cryotraps. The pre-column connects C-2 with the analytical column, and is installed to prevent the column from being exposed to the wide temperature swings that occur at the trap. After surrogates have been introduced on a sample cartridge, the sample is then thermally desorbed by heating the oven while

Table 4: Target Compounds Analyzed for Calibration

Compound	Quantitation Ions
benzene	78
bromodichloromethane	83
carbon tetrachloride	117
chloroethane	64
chloromethane	50
dibromomethane	174
1,1-dichloroethane	63
1,2-dichloroethane	62
1,1-dichloroethene	61
trans-1,2-dichloroethene	61
ethylbenzene	91
m-ethyltoluene	120
methylene chloride	84
styrene	104
1,1,2,2-tetrachloroethane	83
tetrachloroethene	166
1,1,1-trichloroethane	97
1,1,2-trichloroethane	97
trichloroethene	130
trichlorofluoromethane	101
trichloromethane	83
toluene	92
vinyl chloride	62
m-xylene	91
o-xylene	91

purging with helium.

The helium transfers the VOCs from the cartridge to the C-1 trap. The sample is then passed through a heated transfer line and cryofocused at C-2, at the front of the pre-column, where it is injected by flash heating. Table 5 summarizes typical desorber conditions. The chromatographic conditions used are those listed in table 6, as modified from U.S. EPA Method 524.2.

An example of the GC/MS Printout is found in figure 4 (appendix A), which includes target and surrogate compounds in elution order.

3.7.4 Tenax/CMS Cartridge Analysis

Handle all Tenax/CMS samples with cotton cloth or gloves and tweezers to avoid contamination. To analyze a cartridge sample, follow these steps.

1. Place the cartridge in the desorb oven, CMS side first, so that it is downflow from the Tenax. Start the thermal desorber going into the purge step. Set the flow at 20 mL/min.

2. During the purge step, inject 10 mL of a 1-ppm mixture of the surrogate standards (bromochloromethane [BCM] and p-bromofluorobenzene [BFB]), onto the Tenax side of each sample cartridge. Lower the purge flow to 5 mL/min, so that the combined flow through the cartridge does not exceed 20 mL/min.

3. After the surrogates have been introduced on the tube and the purge cycle has been completed, the first cryogenic trap (C-1) is cooled with liquid nitrogen to -160°C. At this time, remove the cartridge, turn it around, and reinsert it into the desorb oven; the Tenax side of the cartridge is now downflow of the CMS.

4. Once the tube has been inverted and C-1 has been cooled, step the thermal desorber to the desorb cycle, allowing the surrogates to desorb from the Tenax and CMS with the sample and flow directly to C-1.

5. At the end of desorb, step the desorber again, cooling the C-2 cryotrap. When C-2 is cooled, the desorber will automatically step to the transfer step, and the sample is cryofocused at C-2.

Table 5: Typical Desorber Conditions

Parameter	Value
Desorb Temperature	240° C
Desorb Time	10.0 minutes (Tenax/CMS only)
Cryotrap-1 (C-1) Temperature	- 160° C
Cryotrap-1 Desorb Temperature	250° C
Transfer (C-1 to C-2)	3.5 minutes
Cryotrap-2 (C-2) Temperature	- 160° C
Cryotrap-2 Desorb Temperature	250° C
Cryotrap-2 Desorb Time	2.0 minutes

Table 6: Chromatographic Conditions

Parameter	Value
Initial Temperature	5.0° C
Initial Time	3.0 minutes
Ramp Rate	8.0° C/minute
Final Temperature	185.0° C
Run Time	25.5 minutes

6. When transfer is complete, the sample will be injected by automatic flash heating of C-2. The analysis then follows the chromatographic conditions in table 6.

3.7.5 Canister Sample Analysis

Canister samples are usually collected at or near atmospheric pressure. To allow the sample to flow from the canister, the canister pressure must be raised above one atmosphere with ultrahigh purity nitrogen. Normally, sample pressure is doubled for ease of calculation.

1. Before attaching the canister sample, purge the pressurizing line of the apparatus with nitrogen as indicated in figure 5 (appendix A). Attach the canister sample to the pressurizing apparatus and close the regulator to the nitrogen cylinder. Open the canister valve, allow the pressure to equilibrate, and record the initial pressure (P_i) in the analysis log.

2. Open the cylinder regulator slowly so the pressure gradually increases. When the canister pressure reaches twice the P_i, close the regulator, then close the canister valve, and record the final pressure (P_f) in the analysis log.

3. Attach the canister to the analysis train at the desorb oven as shown in figure 6 (Appendix A). With the mass flow controller valve closed, open the canister valve to allow the sample to come to equilibrium in the sample train.

4. Start the thermal desorber, and step through

the purge step to the step that cools C-1. When the desorber steps to desorb, lower the flow to zero. Open the mass flow controller valve and begin timing sample flow. The controller flow rate and the desorb time needed for the sample to flow are calculated based on the sample volume required and the equations in section 3.8.

5. Close the canister valve after the precise amount of desorb time has elapsed. Close the mass flow controller valve after the analysis train pressure reaches zero.

6. Replace the desorb oven cover attached to the canister analysis train with the desorb oven cover used for Tenax/CMS samples. Raise the helium flow to 5 mL/min, and inject 10 mL of the surrogate standards while still in desorb. At the end of desorb, follow the analysis procedure in section 7.4, steps 5 and 6.

3.7.6 Analysis of Canister Samples Adsorbed on Cartridges

Canister samples are adsorbed on Tenax/CMS cartridges when the samples are suspected of containing high levels of carbon dioxide or other permanent gases that would freeze the cryotraps.

1. Follow the procedure in section 3.7.5, steps 1 and 2, for the pressurization of the canister sample.

2. Place a Tenax/CMS cartridge in the desorb oven with the CMS side in first. Attach the canister to the analysis train as shown in figure 7 (appendix A).

3. With the mass flow controller valve closed, open the canister valve to allow the sample to come to equilibrilium in the sample train.

4. Start the thermal desorber into the purge step. Lower the purge flow to zero. Open the mass flow controller valve and let the desired sample volume adsorb onto the cartridge.

5. After the sample has been adsorbed, close the canister and mass flow controller valves, replace the desorb oven cover, and inject 10 mL of the surrogate standards while still in the purge step.

6. After surrogates have been spiked on the cartridge, step the desorber to cool C-1, and follow the Tenax/CMS analysis procedure in section 3.7.4, steps 3 through 6.

3.8 CALCULATIONS

Concentrations of target compounds are calculated by the GC/MS computer software. To establish concentration limits that the GC/MS can measure, limits of quantitation (LOQ) are calculated for each sample. LOQs are calculated by the following:

$$LOQ = \frac{(LCV)\ (SC)}{SV}$$

where:

LCV = lowest calibration volume
SC = standard concentration
SV = sample volume (in milliliters)

LOQ varies inversely with the sample volume, and can range from 500 ppb for a minimal sample volume of 5 mL, to as low as 0.1 ppb for a 25-L sample.

When the canister pressure is increased, the dilution factor (DF) is calculated by the following:

$$DF = \frac{P_f}{P_i}$$

where:

P_f = canister pressure (psi) after pressurization,
P_i = canister pressure (psi) before pressurization

The following equation calculates the desorb time necessary for a given sample volume and flow rate:

$$DT = \frac{(SV)\ (DF)}{FR}$$

where:

DT = desorb time (in minutes)
SV = sample volume (in milliliters)
DF = dilution factor (usually 2)
FR = flow rate (in mL/min)

For example, with a DF of 2 and a flow rate of 40 mL/min, it would take 5 minutes to desorb 100 mL of unpressurized sample (equivalent to 200 mL of pressurized sample). For larger sample volumes, it may be necessary to set the thermal desorber for

longer than 10 minutes to desorb the sample and allow time for surrogate spiking.

3.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following quality assurance/quality control procedures apply:

- Two criteria must be satisfied to verify the identification of a target compound:
  - Retention Time - A sample component's retention time (RT) must be within ± 0.50 minutes of the RT of the standard component. For reference, the standard must be run on the same day as the sample.
  - Spectra - (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (where the most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. (2) The relative intensities of the ions specified above must agree within ± 20% between the sample and the reference spectra.
- The GC/MS is tuned daily for PFTBA to meet the abundance criteria for BFB as listed in U.S. EPA Method 624. The tune is adjusted when necessary.
- An acceptable three-to-five point calibration of the standards must be run before the analysis. A calibration is acceptable if the Relative Standard Deviation is <25% of the average response factors for each compound. Samples are quantitated on the average response factors of the calibration range.
- A continuing calibration standard must be run for each day of analysis. Standards are checked against the average response factors of the calibration range; if any standard component varies by greater than 25% of the average response factor, re-run the continuing calibration. If the second continuing calibration has components varying by greater than 25% of the average response factor, run a new initial calibration.
- A surrogate standard of BFB and BCM is added to all standards and samples. Percent recoveries for samples are calculated against daily standards. Recoveries should be within 70% to 130% for BFB and BCM.
- Method blanks are analyzed after a standard analysis to check for carryover, and are also necessary after analyzing samples with high levels of contamination. For Tenax/CMS samples, a method blank is an analysis of a new cartridge spiked with surrogates. For canister samples, a method blank is flowing the same volume of nitrogen as the samples into the desorber, followed by surrogate spiking. For canister samples adsorbed onto cartridges, a method blank is a volume of nitrogen equal to the sample volumes adsorbed on a cartridge, followed by surrogate spiking and analysis.
- Ten percent of all samples received are to be analyzed in replicate.
- Performance Evaluation (PE) canisters containing known concentrations of VOCs should be analyzed at least once per analysis for canister samples. The analytical procedure is the same for canister samples.

3.10 DATA VALIDATION

Review of the data generated should be conducted according to the Quality Assurance/Quality Control considerations listed in section 3.9.

3.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and laboratory health and safety practices.

4.0 PREPARATION OF SUMMA CANISTER FIELD STANDARDS: SOP #1706

4.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the preparation of SUMMA canister field standards. SUMMA polished canisters are used to store calibration gas standards for transport to field sampling sites. These standards will be used for calibrating field instruments. In addition, a series of different concentrations of gas standards, or dilutions in the field of a single canister, can be used to construct calibration curves and to ascertain minimum detection limits on various field instrumentation currently used by EPA/ERT.

4.2 METHOD SUMMARY

A certified gas standard cylinder is selected and set for delivery pressure of 20-30 psig. The hoses are bled with the gas standard. Then, a clean, evacuated SUMMA canister is attached to the gas standard line and is opened and charged to 20-30 psig with the certified gas standard cylinder. The SUMMA canister is closed and the gas standard lines are removed. A "tee" with a septum is attached onto the Swagelok fitting of the SUMMA canister. The "tee" is purged with the contents of the SUMMA canister. The SUMMA canister valve is opened and samples are taken via a gas-tight syringe through the septum on the "tee." When not in use, the valve is closed. Tedlar bags can also be filled from the "tee."

4.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples and gas standards can be kept several months in the SUMMA polished canisters. Care must be taken to ensure no leaks occur when the "tee" and septum are used. In addition, the needle valve on the SUMMA canister must be completely closed when not in use. When transporting and storing, the SUMMA canister is placed in a plastic shipping container. This will protect the canister from accidental punctures or dents.

4.4 INTERFERENCES AND POTENTIAL PROBLEMS

As long as the gas standards and all transfer lines are clean, no interferences are expected. The initial pressure of the SUMMA canister should be recorded after filling. In addition, the pressure should be recorded after each use. A dramatic drop in pressure (e.g., 5 psig or more) may invalidate the use of that canister.

4.5 EQUIPMENT/APPARATUS

- SUMMA canister, 6-liter total volume. While there may be other sources, two readily available sources are Cat. # 87-300, Anderson Samplers, Inc. 4215 Wendell Drive, Atlanta, GA 30376; PN # 0650, SIS, P.O. Box 8941, 815 Courtney St., Moscow, Idaho 83843.
- certified gas standard from Scott Gas, Matheson or other reliable manufacturer.
- Hamilton gas-tight syringe with Teflon-seal plugs in various sizes.
- clean Teflon tubing, 1/4-inch OD.
- Teflon Swagelok "tee," 1/4-inch OD.
- 1/4-inch Teflon Swagelok nuts and ferrules.
- 9-mm Septa, preferably Teflon backed.
- stainless steel Swagelok on/off or needle valve, 1/4-inch OD.

4.6 REAGENTS

All standards must be vapor-phase pressurized gas cylinders, certified by the manufacturer to be within ±2% accuracy, and to be National Bureau of Standards (NBS) traceable. Scott Specialty Gas or Matheson Gas can provide these standards. If field

dilution is required, a cylinder of ultrahigh purity air is required.

4.7 PROCEDURES

1. Obtain a SUMMA polished canister that has been cleaned and evacuated as per ERT SOP #1703 (SUMMA Canister Cleaning) and select a compressed-gas cylinder of a certified standard. This standard should be certified by the manufacturer to be within ±2% accuracy of the concentration level and be NBS traceable.

2. Attach a high-purity, dual-stage regulator to the standard cylinder. This must deliver 20-30 psig pressure at an accuracy of ±10% or better.

3. Attach a section of clean, unused 1/4-inch OD Teflon tubing to the Teflon "tee." The side port of the "tee" has an on/off valve or needle valve connected to it (see figure 8, appendix A).

4. Temporarily connect a vent line to the outlet port of the side valve and vent it to a fume hood or to an outside vent. The SUMMA canister charging system appears in figure 9, appendix A.

5. Open the standard cylinder to 20-30 psig at the outlet of the cylinder regulator.

6. The needle valve on the SUMMA canister is still closed at this point. Open the side valve on the "tee" and allow the standard cylinder's 1/4-inch Teflon feed lines to vent for 1 to 2 minutes.

7. Then close the valve tightly and slowly open the needle valve on the SUMMA canister. A hissing noise should be heard. Allow the canister to continue filling. Do not fill the SUMMA canister too rapidly.

8. Periodically check the pressure on the dual stage regulator attached to the standard cylinder to ensure 20-30 psig is being delivered.

9. Once the hissing stops, the canister should be filled to approximately the same pressure as that of the source line.

10. Close the needle valve on the SUMMA canister tightly.

11. Close the standard cylinder and vent the feed lines.

12. Remove the feed line from the top of the Teflon "tee."

13. Place a Swagelok back ferrule, in the inverted position, on the top of the "tee". This will provide a flat surface on which a Teflon-backed septum can be placed.

14. Place the Teflon-backed septum, Teflon side down. The septum should create a gas-tight fit once a 1/4-inch Swagelok nut is tightened onto the top of the "tee" (see figures 10 and 11, appendix A).

15. Open the needle valve on the SUMMA canister to check for leaks throughout the "tee", particularly in the septum fitting. Do this with the valve on the side of the "tee" closed.

16. Afterwards, slowly open the side valve of the "tee" and vent for 1/2 minute and re-close. The septum "tee" is now ready for sampling from the canister using a gas-tight syringe through the septum seal.

17. Close the SUMMA canister needle valve between sample taking with the gas-tight syringe.

18. Periodically, vent or flush the "tee" to provide fresh standard for sampling. The side valve can also be used, after flushing, to fill Tedlar bags with the standard from the SUMMA canister.

4.8 CALCULATIONS

The procedure for performing field dilutions of the standards from the SUMMA canisters must be documented. This allows for the recalculation of concentrations of standards if any discrepancies arise in the calibration of the field instrumentation. Simple volumetric dilutions using Hamilton gas-tight syringes are performed using Tedlar bags with ultra-high purity air as the diluent.

4.9 QUALITY ASSURANCE/ QUALITY CONTROL

The concentration levels of the certified gas standards must be recorded. The vendor typically provides the analysis of certification with each standards cylinder; a copy should be provided with the SUMMA canister.

As previously stated, the pressure of the canister along with the date and time, should be recorded at the initial filling and at the end of each use of the canister. A drop in pressure of 5-10 psig between usages may invalidate the canister for use as a calibration standard. Certification of canister cleaning and evacuation should be noted prior to filling with standards.

4.10 DATA VALIDATION

This section is not applicable to this SOP.

4.11 HEALTH AND SAFETY

Pressurizing of SUMMA canisters should be performed in a well-ventilated room, or preferably under a fume hood. Care must be taken not to exceed 40 psig in the canisters. Canisters are under pressure, albeit only 20-30 psig, and should not be dented or punctured. They should be stored in a cool, dry place and always be placed in their plastic shipping boxes during transport and storage.

5.0 LOW LEVEL METHANE ANALYSIS FOR SUMMA CANISTER GAS SAMPLES: SOP# 1708

5.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) is intended for use when analyzing SUMMA canister gas samples for low parts per million volume (ppmv) levels of methane.

5.2 METHOD SUMMARY

A flame ionization detector (FID) gas chromatograph (GC) is used to separate and quantitate methane in gas samples. The sample is introduced into the carrier gas as a plug and passes through a gas chromatography column, which then separates it into two peaks. The first peak is unresolved air; the second peak is resolved methane. Peak areas are used in conjunction with calibration plots for quantitative measurements. This separation is completed in 5 minutes.

5.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Refer to U.S. EPA Method T014 concerning SUMMA canister cleaning and sample collection. In addition, refer to ERT SOP #1703, SUMMA Canister Cleaning and ERT SOP #1704, SUMMA Canister Sampling.

Canisters are stored and analyzed at room temperature.

5.4 INTERFERENCES AND POTENTIAL PROBLEMS

This section is not applicable to this SOP.

5.5 EQUIPMENT/APPARATUS

- gas chromatograph -- Varian 3400 gas chromatograph with flame ionization detector (or equivalent) capable of operating at 225°C.
- carrier gas cylinder -- ultrahigh purity helium with a two-stage regulator delivering a pressure of 90 psi.
- 1 mL and 0.1 mL precision gas-tight syringes with needles for sample introduction.
- gas chromatography column -- 10 feet by 1/4 inch stainless steel column packed with Spherocarb, 100/120 mesh (or equivalent), capable of operating at 100°C, as well as injection temperatures of 200°C.
- electronic integrator -- Spectra-Physics SP4290 integrator (or equivalent).
- septum port adaptor for SUMMA canister.
- soap film flow meter (or equivalent).

5.6 REAGENTS

- helium -- ultrahigh purity grade helium (99.9999%).
- hydrogen -- ultrahigh purity grade hydrogen (99.9999%).
- air -- ultrazero air (<0.05 ppmv total hydrocarbon).
- calibration standards (in the range of 5-100 ppmv) -- methane standards, balance air.

5.7 PROCEDURES

5.7.1 Gas Chromatograph

1. Turn the carrier gas on and adjust the flow rate to 40 mL per minute.
2. Turn the air on and adjust the flow rate to 150 mL per minute.

3. Turn the hydrogen on and adjust the flow rate to 30 mL per minute.

4. Check the flows with a soap film flow meter.

5. Ignite the flame ionization detector and allow it to equilibrate for 10 minutes.

6. Turn the integrator on and zero it before samples are introduced.

5.7.2 Calibration

1. Introduce, via 1-mL syringe, aliquots (of the same size as will be used on the sample injections) of the standard calibration gas mixtures into the gas chromatograph injector. At least one injection of each standard gas mixture is required before starting to analyze samples. Perform the very first calibration in triplicate.

2. Verify the initial calibration by injecting a complete set of at least four standards (at least five different concentrations of standards are routinely available from commercial suppliers) at the beginning of each day's analytical activities. It is suggested that each sample injection be followed systematically by a standard injection so that many injection areas are tabulated and averaged in the report.

5.7.3 Injection of Sample

1. Withdraw a 1-mL sample from the SUMMA septum port using a 1-mL gas-tight syringe.

2. Quickly inject the sample, guarding against blow-back of the plunger. Simultaneously, activate the integrator and label the sample run.

3. End the integrator run in 5 minutes and re-zero before the next analysis.

Samples analyzed above the calibrated linear range can be reanalyzed by injecting a smaller volume, or by diluting in ultrahigh purity zero air to acquire responses within the linear range. These dilutions may be done by injecting a measured volume of the sample into a Tedlar bag and adding a measured volume of zero air. For instance, 100 mL of sample measured with a gas-tight syringe, added to 900 mL of zero air, would be diluted by a factor of 10. These volumes have to be recorded and taken into account in the calculations.

5.8 CALCULATIONS

Prepare a linear standard curve of ppmv versus peak area. Calculate the sample concentrations using the formula $y = mx + b$; where y is the peak area, m is the slope (peak area/ppmv), b is the y intercept (peak area), and x is the concentration (ppmv).

The above equation may be rearranged to:

$$x = y - \frac{b}{m}$$

where y is measured area, corresponding to a sample injection and x is the desired methane concentration in the sample injection. If a dilution has been made then, of course, the concentration obtained must be multiplied by the ratio of the final sample volume to the initial sample volume. Most integrator packages will handle the above calculations but it is recommended that a commercial spreadsheet program be used.

5.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following quality assurance/quality control procedures are applicable.

5.9.1 Precision

The precision of the method is monitored during the second lowest calibration standard from the linear curve. A control range is established for the standard using three standard deviations from the mean of 10 independent analyses. The standard is analyzed periodically (at the beginning and end of a series of samples or every 8 hours) and must respond within the range of three standard deviations for the system and data precision to be considered under control. If the results of the standard analysis are out of range, the system must be repaired and the standards rerun, or a new calibration curve must be performed.

5.9.2 Accuracy

The accuracy of the method is monitored by periodically analyzing blind performance evaluation samples. These samples should not be prepared by the same outside source which provided the calibration standards.

5.10 DATA VALIDATION

Data will be evaluated based on the information provided in section 5.9.

5.11 HEALTH AND SAFETY

When working with potentially hazardous materials, refer to U.S. EPA, OSHA and site-specific health and safety practices.

6.0 ASBESTOS SAMPLING: SOP #2015

6.1 SCOPE AND APPLICATION

The objective of this Standard Operating Procedure (SOP) is to outline a method for sampling asbestos fibers in indoor and outdoor/ambient air at hazardous waste sites.

Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M).

Both 40 CFR 763 and its addendum provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements.

The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing for handling asbestos. Also, these regulations specify:

- The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cm^3 of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 μm.

- An action level of 0.1 fibers/cm^3 (one-half the OSHA standard) is the level U.S. EPA has established at which employers must initiate such activities as air monitoring, employee training, and medical surveillance.

References to specific analytical methodologies are made throughout this document. Also, be aware that EPA is developing an Environmental Asbestos Assessment Manual. An interim draft document titled "Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method" (May 1990) is available and recommended for use as the most current method.

6.2 METHOD SUMMARY

Asbestos has been used in many commercial products including such building materials as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalt. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers.

Asbestos air sampling is conducted by drawing air through a filter at a known flow rate with a flow-controlled pump. The sample is then analyzed using Phase Contrast Microscopy (PCM) and/or Transmission Electron Microscopy (TEM).

PCM analysis is widely available and is less costly than TEM. TEM is considered the best method for identifying airborne asbestos. TEM can detect very thin fibers typically down to 0.0025 μm in diameter.

When TEM-produced data (U.S. EPA) is compared with data from PCM (NIOSH), the TEM's aspect ratio of 5 to 1 should be modified to 3 to 1.

6.2.1 Pump Calibration

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette." A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before and after collecting the sample. Should the flow rate change by more than 5% during the sampling

period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. Sampling pumps can be calibrated prior to coming on site so that time is saved when performing onsite calibration.

Personal sampling pumps are utilized when the flow rates are between .001 L/min to 5 L/min. Many lightweight portable pumps are capable of providing high or low volume air flow. See the manufacturer's manual for pump operation.

High-flow pumps are utilized when flow rates between 4 L/min to 16 L/min are required. High-flow pumps are used for short sampling periods to obtain the desired sample volume. ERT uses the Gilian Aircon 520. An equivalent high-flow pump can also be used.

High-flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available, then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several electrical extension cords may be required if sampling locations are remote.

6.2.2 Outdoor/Ambient Sampling

PCM analysis may be used for outdoor/ambient air samples. When analysis shows total fiber count above the EPA action level of 0.1 fibers/cm^3 of air, then TEM can be used to identify asbestos from non-asbestos fibers. Some labs are able to perform PCM and TEM analysis on the same filter, however, this should be verified with the laboratory prior to analysis.

High-volume pumps, for the most part, are used for outdoor sampling in low dust areas. The samplers should be placed above ground level, about 4 to 5 feet high, away from obstructions that may influence air flow. Table 7 summarizes outdoor sampling locations and the rationales for their selection.

Outdoor sampling usually requires flow rates between 10 to 15 L/min with a sample volume of 1000 to 5000 liters. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

It is recommended that a meteorological station be established. If possible, sample after 2 to 3 days of dry weather and when the wind conditions are at 10 mph or greater.

6.2.3 Indoor Sampling

EPA uses PCM analysis for indoor air samples. When analysis shows total fiber count above the EPA action level of 0.1 fibers/cm^3 of air, then TEM can be used to identify asbestos from nonasbestos fibers.

Sampling pumps should be placed 4 to 5 feet above ground level, and away from obstructions that may influence air flow. The pump can be placed on a table or counter. Table 8 summarizes indoor sampling locations and the rationales for their selection.

Indoor sampling generally utilizes high-flow rates and increased sample volumes in order to obtain lower detection limits, i.e., 0.01 fibers/cm^3 of air or less (with PCM) and 0.005 structures/cm^3 or less (with TEM).

6.2.4 Aggressive Sampling

Sampling equipment at fixed locations may fail to detect the presence of asbestos fiber. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option characterize the worst-case condition. This is referred to as aggressive air sampling for asbestos.

6.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

6.3.1 Filter Selection and Collection Device

Which filter and collection device to use for sample collection will depend upon which analytical methodology is utilized.

Table 7: Sampling Stations for Outdoor Sampling

Sampling Station Location	Procedure	Rationale
Upwind/Background	Collect a minimum of 2 simultaneous upwind/background samples 30° apart from the prevailing windlines	Establishes background fiber levels
Downwind	Deploy a minimum of 3 sampling stations in a 180° arc downwind from the source	Indicates if asbestos is leaving the site
Site Representative and/or Worst Case	Obtain one representative sample which shows average on-site conditions or obtain worst-case sample (optional)	Verify, continually confirm, and document selection of proper levels of worker protection

Note: More than one background station may be required if the asbestos originates from different sources.

Table 8: Sampling Stations for Indoor Sampling

Sampling Station Location	Procedure	Rationale
Indoor Sampling	• If a work site is a single room, disperse five samplers throughout the room • If the work site contains up to five rooms, place at least one sampler in each room • If the work site contains more than five rooms, select a representative sample of the rooms	Establishes representative samples from a homogeneous area
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines	Establishes whether indoor asbestos concentrations are coming from an outside source
Worst Case	Obtain one worst-case sample by aggressive sampling (optional)	Verify, continually confirm, and document selection of proper levels of worker protection

- NIOSH Method 7400: Phase Contrast Microscopy involves using a 0.8 to 1.2 μm cellulose ester membrane, 25-mm diameter, 50-mm conductive cowl on cassette (figure 12, appendix A).

- U.S. EPA Transmission Electron Microscopy involves using a 25-mm filter cassette with either a polycarbonate filter having a pore size <0.4 μm or mixed cellulose ester filter (MCE) having a pore size <0.45 μm. This cassette includes an extension cowl, a 5.0 μm MCE backup filter to serve as a diffuser, and a support pad (figure 13, appendix A).

6.3.2 Sample Handling Procedures

1. Place a sample label on the cassette with a unique sampling number. Do not put sampling cassettes in your shirt or coat pockets as the filter can pick up fibers. ERT uses the original cassette box to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Mark each bag to indicate sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is at the bottom. Use enough packing material to prevent jostling or damage. If possible, hand carry to laboratory.

4. Provide appropriate documentation with samples (e.g., chain-of-custody form and requested analytical methodology).

5. Follow all QA/QC requirements from the lab as well as from the PCM/TEM analytical methodology (e.g., field blank and lot blank requirements).

6.4 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates should not exceed 16 L/min due to the possibility of asbestos fiber disintegration upon contact with the filter.

6.4.1 NIOSH Method 7400, PCM

- PCM cannot always distinguish asbestos from non-asbestos fibers. All particles meeting the counting criteria are counted as total asbestos fibers.

- Fibers less than 0.25 μm in length will not be detected by this method.

- High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

6.4.2 U.S. EPA's TEM Method

- High concentrations of background dust interfere with fiber identification.

6.5 EQUIPMENT/APPARATUS

6.5.1 Personal Sampling Pump

- personal sampling pump (e.g., Gilian Personal Sampler)
- inert tubing with glass cyclone and hose barb
- sampling cassettes with conductive cowl.
- appropriate membrane filters.
- rotameters
- whirlbags for cassettes
- tools -- small screw drivers
- sample labels
- air data sheets
- container -- to keep samples upright

6.5.2 High-Flow Pump

- high-flow pump (e.g., Gilian Aircon)
- generator or electrical outlet
- extension cords
- rotameters
- inert tubing -- unless provided with pump
- sampling cassettes with conductive cowl
- appropriate membrane filters
- whirlbags for cassettes
- sample labels
- air data sheets
- container -- to keep samples upright

6.6 REAGENTS

This section is not applicable to this SOP.

6.7 PROCEDURES

6.7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and what supplies and equipment are needed.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or preclean equipment, and ensure that it is in working order.

4. Prepare schedules, and coordinate with staff, client, and regulatory agency, as appropriate.

5. Perform a general site survey prior to entry in accordance with the site-specific health and safety plan.

6. Use stakes or flagging to identify and mark all sampling locations.

6.7.2 Aggressive Sampling

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should last at least 5 minutes per 1000 square feet of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Start the sampling pumps and sample for the required time.

4. Turn off the pump and then the fan(s) when sampling is complete.

6.7.3 Personal Sampling Pump

1. Charge the unit for the maximum required time as indicated in the manufacturer's manual.

2. In the clean zone of the site, follow the calibration procedures in section 6.9.1 to 6.9.3.

3. Mobilize to the sampling location.

4. To set up the sampling train, attach one end of the polyvinyl chloride (PVC) tubing (approximately 2 feet) to the cassette base; attach the other end of the tubing to the inlet plug on the pump (figure 14, appendix A). The attachment between the cassette base and the tubing can best be achieved by using a hose barb with a cyclone clip.

5. Place the sampling pump 6 feet above ground level (in the breathing zone) and in an area that will not be affected by unusual air flow. The sampling pump and cassette can be placed on a sturdy structure, attached to a dowel rod or hooked to an object.

6. Remove the cassette cap from the extension cowl (open faced) and orient the cassette perpendicular to the wind.

7. Adjust the time on the pump. If the pump is programmable, turn past the zero mark before setting the actual time.

8. Turn the pump on.

9. Record the following in the site logbook: date, time, location (area or room), sample identification number, pump number, flow rate and desired total sampling time.

10. Record weather data (e.g. ambient temperature, wind direction, windspeed, precipitation).

11. Check the pump at midpoint of the sampling period if longer than 4 hours.

12. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

13. At the end of the sampling period, check the fault button to obtain pump sampling time. (This indicates whether or not the pump ran the full programmable timespan). Be sure to orient the cassette in an upright position to prevent fibers from falling from the filter when the vacuum is released.

14. Record the pump run time (finish time minus start time).

15. Perform post-calibration procedures as shown in section 6.9.

16. Record the post-flow rate in a field logbook.

17. Remove the PVC tubing from the sampling cassette. While holding the cassette upright, replace the inlet plug on the cassette cap.

18. Place the outlet plug on the cassette base.

19. Refer to section 6.3.2, steps 1-5 for sample handling procedures.

6.7.4 High-Flow Pump

The following instructions are for a Gilian Aircon 520 Constant High-Flow Air Sampler and is used for illustrative purposes; an equivalent high-flow pump can be used instead.

1. Once on site, perform the calibration in the clean zone. The calibration procedures for personal sampling pumps listed in section 6.9.1 are also applicable to high volume sampling pumps.

2. After calibrating the high volume sampler, mobilize to the sampling location.

3. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap. The cassette should be positioned perpendicular to the wind (figure 15, appendix A).

4. Turn the generator on. The generator should be placed 10 feet downwind from the sampling pump.

5. Record the pump's cumulative time (if applicable).

6. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

7. Record weather: wind speed, ambient temperature, wind direction, and precipitation.

8. Turn the pump on.

9. Check the pump at sampling midpoint if longer than 4 hours.

10. At the end of the sampling period, orient the cassette up, and turn the pump off.

11. Record the cumulative time (if applicable).

12. Check the flow rate as shown in section 6.9. The sampling cap is replaced before calibrating.

13. Record the post-flow rate.

14. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

15. Refer to section 6.3.2, steps 1 to 5, for sample handling procedures.

6.7.5 Calibration

An electronic calibrator is used for calibrating rotameters and pumps. Refer to section 6.9.1 to 6.9.3 for calibration procedures.

6.8 CALCULATIONS

The sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, table 9 contains suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor readings in mg/m^3.

PCM utilizes flow rates between 0.5 L/min and 16 L/min. The sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 hours is appropriate in non-dusty atmospheres containing 0.1 fibers/cm^3. Dusty atmospheres (areas with high levels of asbestos) require smaller sample volumes (<400 L) to obtain countable samples. In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high-flow rates (7 to 17 L/min) over shorter sampling times. In relatively

Water Sample Analysis

1. Use a pipette to place a 20-mL aliquot of sample into a clean 40-mL VOA vial. Seal the vial with a Teflon-lined septum screw cap.

2. Shake the capped vial vigorously by hand for 1 minute. Allow it to stand, inverted and undisturbed, for at least 30 minutes at ambient temperature for vapor phase equilibration.

3. Use a gas-tight syringe to extract an aliquot of headspace by inserting the syringe needle through the vial septum to a distance approximately halfway between the liquid surface and the septum's Teflon face.

4. Purge the syringe barrel three to five times by withdrawing and expelling a volume of headspace in slight excess of the volume anticipated to be used for analysis.

5. If sample concentrations are high, injection volumes may be reduced to obtain on-scale response. If sample headspace injection volume is reduced below the volume of the aqueous calibration standard used to establish the method detection limit (MDL), the detection limit for target compounds detected at the reduced headspace volume must be determined. Do this by injecting headspace aliquots at the reduced volume into the GC, beginning with the 10-ppb calibration standard and increasing or decreasing standard concentrations, as warranted, until a response for all target compounds has been obtained. The detection limit for parameters detected at the lower headspace injection volume is then calculated using the equation in Section 6.7.1.

6. Identify the compounds in the sample by comparing the RTs of the peaks in the sample chromatogram with those in standard chromatograms. The width of the retention time windows used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of an RT can be used to calculate a suggested window size; however, the judgment of the analyst should be a major factor in the interpretation of chromatograms.

Soil Sample Analysis

1. Place a clean, empty, 40-mL glass vial on the balance. Zero the balance. Using a clean stainless steel spatula, add 5.0 g ± 0.1 g of soil sample.

2. Using a pipette, place enough reagent water in the vial to bring the total volume of the soil and water to 20 mL. Seal the vial with a Teflon-lined septum screw cap.

3. Shake the capped vial vigorously for 1 minute, to promote dispersion of the soil sample and increase surface area. Allow to stand, undisturbed, at ambient temperature for at least 1 hour for vapor phase equilibrium.

4. Use a gas-tight syringe to extract an aliquot of headspace by inserting the syringe needle through the vial septum to a distance approximately halfway between the slurry surface and the septum's Teflon face. Although 5 g is recommended for most soil matrices, other amounts ranging from 1 g to 10 g have also been used, depending on sample concentrations and the consistency of the matrix.

5. Purge the syringe barrel three to five times by withdrawing and expelling a volume of headspace in slight excess of the volume anticipated to be used for analysis. Wipe the syringe needle with a Kimwipe before injection into the GC.

6. If sample concentrations are high, either reduce the injection volumes or analyze less soil to obtain on-scale response. If sample headspace injection volume is reduced below the volume of the aqueous calibration standard used to establish the MDL, follow the procedure in Section 6.7.1 to determine the MDL for target compounds detected at the reduced headspace volume. Alternatively, weigh out as little as 1 g, keeping in mind that this means a 10% error if using a portable balance accurate to ± 0.1 g.

7. Identify the parameters in the sample by comparing the retention times (RTs) of the peaks in the sample chromatogram with those in standard chromatograms. The width of the retention time windows used to make identifications should be based on

measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of an RT can be used to calculate a suggested window size; however, the judgment of the analyst should be a major factor in the interpretation of chromatograms.

6.8 CALCULATIONS

6.8.1 Air and Soil Gas Samples

Determine the concentration of individual compounds in each sample by using:

$$[Sample] = [Std] \frac{A_1}{A_2} \cdot \frac{V_2}{V_1}$$

where:

sample	=	concentration of sample in ppb or ppm
A_1	=	peak area of sample (volts × seconds)
A_2	=	peak area of standard (volts × seconds)
V_1	=	injection volume of sample (μL)
V_2	=	injection volume of standard (μL)
std	=	concentration in ppm or ppb

6.8.2 Water Samples

Determine the concentrations of individual compounds in the sample by using:

$$*[Sample] = [Std] \frac{A_1}{A_2} \cdot \frac{V_2}{V_1}$$

where:

sample	=	concentration of sample in ppb or ppm
A_1	=	peak area sample (volts × seconds)
A_2	=	peak area standard (volts × seconds).
V_1	=	injection volume of sample (μL)
V_2	=	injection volume of standard (μL)
std	=	concentration in ppb or ppm

*This is the same as Equation 3, except the concentration of the headspace standard is used.

6.8.3 Soil Samples

Determine the concentrations of individual compounds in the sample by using:

$$[Sample] = [Std] \cdot \frac{A_1}{A_2} \cdot \frac{V_2}{V_1} \cdot \frac{V_{hdsp}}{W_{samp}}$$

where:

sample	=	concentrations in μg/kg (ppb)
A_1	=	peak of area sample (volts × seconds)
A_2	=	peak of area standard (volts × seconds)
V_2	=	injection volume of standard (μL)
V_1	=	injection volume of sample (μL)
V_{hdsp}	=	volume of headspace (always 20 mL)
W_{samp}	=	weight of sample (usually 5 g)
std	=	concentration in μg/kg (ppb)

6.9 QUALITY ASSURANCE/ QUALITY CONTROL

In order to meet the QA2 data quality objectives, at least 10% of all field samples must be confirmed by GC/MS analysis. The following QA/QC requirements must be followed and provided in the data package submitted:

- Chain of custody documentation;
- Sample log -- date/time of sample collection, date/time of analysis, and run numbers;
- Blanks (see Section 6.9.1);
- Instrument calibration data;
- Labeling of chromatograms -- identify each chromatogram clearly by analysis type (i.e., syringe blank, sample number, or calibrant concentration), injection volume, run number, date, and time;
- Replicate sample analysis -- after every ten samples to check method/analyst precision. The RSD of the area response of any of the compounds should be within 15%;

- Retention time/instrument response check -- since compound identification is based upon retention time matches, run a calibration standard after every 10-15 samples;

- Spikes -- soil and water samples only (see Section 6.9.2); and

- Confirmational analysis for air and soil gas (see Section 6.9.3).

6.9.1 Blanks

- Air and soil gas analysis -- for each day of analysis, field standards (Tedlar bags filled with gas standards) and field blanks (Tedlar bags filled with ultrazero air) accompany samples through collection, handling, and storage.

- Water analysis -- field blank; duplicate 40-mL VOA vials completely filled with reagent water accompany each cooler used for sample collection, storage, and/or shipment.

- Soil analysis -- reagent blank; 20 mL of the reagent water used in the soil analysis is placed by pipette into a clean 40-mL VOA vial, allowed to equilibrate, and analyzed before any samples.

- Syringe blanks -- syringe blanks are to be run prior to each sample analysis. In practice, there is no need to run syringe blanks if the previous sample is clean.

6.9.2 Spikes

- For every 20 (soil or water) samples, one matrix spike (MS) and one matrix spike duplicate (MSD) must be analyzed. If there are less than 20 samples in a matrix, at least one MS/MSD must be analyzed.

- The samples spiked should have moderate concentrations, if possible. The amount spiked should be equivalent to the middle of the calibration range or one to five times the sample background concentrations, whichever is higher.

- Calculate the percent recovery (%R) of each compound of interest from:

$$R = 100 \cdot \frac{(A-B)}{S}$$

where:

R = percent recovery
A = concentration of sample and spike
B = concentration of sample
S = concentration of the spike

- The percent recovery (%R), should be 50-90% for a soil matrix, and 80-120% for a water matrix. Due to the complexity of the soil/water/vapor equilibria, recoveries from soil matrices are consistently below 100%.

6.9.3 Confirmatory Analysis

Depending on work plan stipulations, at least 10% of the air and soil gas samples analyzed by this GC method must be submitted for confirmatory GC/MS analysis according to modified methods TO-1 (Tenax absorbent) and TO-2 (Carbon Molecular Sieve [CMS] absorbent). Each soil gas sample must be absorbed on replicate Tenax/CMS tubes. The volume absorbed on a Tenax/CMS tube is dependent on the total concentration of the compounds measured by the Photovac as shown below:

Total Concentration	Sample Volume (mL)
> 10	use serial dilution
10	10 - 50
5	20 - 100
1	100 - 250

A range of volumes is given to account for sample variability. The low end of the range should be used for samples whose total concentrations are primarily one large peak, since too large a volume will overload the GC/MS column when that peak is confirmed. The high end of the range should be used for multi-peak samples.

6.10 DATA VALIDATION

Data should be reviewed to ensure that the QA/QC requirements listed above have been met.

6.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety practices. More specifically, the samples should be stored in a cooler away from the analysis area, if possible. The analysis area should have adequate ventilation.

7.0 MICROMONITOR M200: SOP #2111

7.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) presents an overview of the Micromonitor M200 dual-channel microchip gas chromatograph (GC), including analytical capabilities, operating methods and technical limitations. This microchip gas chromatograph, complete with high resolution capillary columns, is linked to a Macintosh personal computer, allowing for rapid field analysis of environmental samples.

The M200 is a portable, high-speed gas chromatograph that samples gases and vapors, separates any volatile components, and then identifies and calculates the concentrations of these compounds. Only vapor-phase samples can be introduced into the M200. Purgeable organics from soil and water matrices are collected via a portable sample concentrator and converted to vapor phase for analysis by the M200. No liquids should ever be introduced into the M200 GC system.

7.2 METHOD SUMMARY

A Macintosh computer system connected to the M200 processes sample data and identifies selected compounds found in vapor-phase samples. The M200 unit contains two high-resolution capillary columns. Simple front panel controls on the unit allow the operator to adjust all method parameters (Figure 2, Appendix D). After the correct parameters are set, a vapor sample is drawn into the unit via the sample port and an internal vacuum pump. The sample gas is then analyzed on one or both capillary columns and specific volatile compounds are detected, separated and identified by the attached computer system.

Calibration standards must be analyzed prior to field sampling. Once the calibration has been validated and the points plotted using the Macintosh library and software, sample analyses can proceed. The Macintosh calibration library currently contains only target compounds historically encountered in U.S. EPA/ERT field work.

7.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Since the M200 is involved with vapor-phase samples, all samples are collected directly or stored in Tedlar gas sampling bags, as per ERT SOP #2050, Tedlar Bag Sampling.

The actual introduction of any calibration or sample gas into the instrument is done by attaching a 2 cm^3 Beckton-Dickerson glass syringe via a Teflon Luer lock valve directly to the sampling inlet port in the front of the unit.

7.4 INTERFERENCES AND POTENTIAL PROBLEMS

Even considering the powerful analytical capabilities in the M200's design configuration, the instrument has several inherent limitations that impact its ability to be used during responses to chemical releases. These limitations are as follows:

- Commercial versions of the device can detect only preselected compounds that can be identified by parameters stored within its internal read-only memory (ROM) library.
- Large quantities of other vapors could be present and may seriously interfere with the analysis using the M200.
- The presence of common fuels containing many individual components, such as gasoline, will confuse the M200's computerized interpretation of data.
- Large quantities of a preselected vapor may overload the capillary columns, and the retention times of the preselected compounds may fall outside of expected retention time windows.
- Commercial versions of the device are programmed with detection limits which are too high (by factors of 10 to 100) for determining levels of hazardous chemicals at or

below TLV concentrations. For typical soil gas analysis, parts per billion (ppb) will be required, and the M200 cannot detect levels that low. To compensate, a sample preconcentrator is used to concentrate the compounds of interest prior to injection.

7.5 EQUIPMENT/APPARATUS

In addition to the basic Micromonitor M200 unit, several optional pieces of equipment are required for its operation. These pieces include a 12-volt power supply, a Macintosh computer system, and a RS232DB9-DB9 cable (Figure 3, Appendix D). An optional sample preconcentrator is required to analyze air samples with detection limits in the parts per billion range. If purgable organics are to be determined in soil or water matrices, the sample preconcentrator is a required piece of equipment.

7.6 REAGENTS

The M200 utilizes high purity (99.995% or above) helium as a carrier gas. Pressure at the two-stage regulator on the tank should be approximately 80 psig. The capillary column head pressure inside the M200 unit should be 15 to 40 psig. This capillary column head pressure can be adjusted via two regulators found on the back of the instrument (Figure 4, Appendix D).

Gas standards are purchased as certified mixtures at fairly high concentrations (i.e., 10 ppm or greater) from Scott Gas or Matheson. These concentrations are for subsequent dilution to various concentrations that enable construction of a standard calibration curve.

Liquid phase standards, if required, must be of the highest purity, such as Aldrich Gold Label or Supelco Environmental standards kits. If air is to be used for sample/standards dilutions, it must also be ultrahigh purity gas.

7.7 PROCEDURES

1. Plug in your M200 unit. The following message should appear on the display reading: "Self test OK."

2. An asterisk (*) should appear in the first character of the display within 3 minutes of turning on the unit, signaling that the unit is ready (i.e., the temperatures are at the setpoints, the pressures are above 5 psig and the system self test found no errors). If the asterisk does not appear, consult the troubleshooting guide in Appendix C.

3. Enter the method parameters by pressing the <METHOD> key. The first item reads "Column Temperature." If column temperature is 30°C, press <ENTER> to accept the value and go on to the next item. If it does not read 30°C, press <UP> or <DOWN> to achieve this required temperature. Once 30°C is shown, press <ENTER> to accept the value and to advance to the next parameter. The temperature can be set at any level between 30° and 180°C. Table 4 gives appropriate values for three often-used GC columns.

4. Continue through all the items in the method the same way.

5. Now read the "Column Head Pressure" on the front panel by pressing <STATUS>. Press <ENTER> once to advance to the next line where the column pressure will be indicated.

6. Adjust the pressure regulator in the rear of the instrument until the pressure reads about 20±5 psig (or the appropriate pressure required for the selected method). Press <STATUS> to monitor column pressure.

7.7.1 Macintosh Software

The procedures used when the M200 unit is being controlled from the computer system are outlined in the Macintosh M200 Chromatography Applications Manual.

Table 4: M-200 Operating Conditions for Various Sample Columns

	Column Type		
Operating Conditions	**DB-5/DB-1701**	**MOL Sieve 5A**	**HAYESEP A**
Column Head Pressure	20 ± 5 psig	12 ± 5 psig	18 ± 5 psig
Column Temperature	30° C	60° C	60° C
Run Time	40 sec	30 sec	40 sec
Sampling Time	5 sec	5 sec	5 sec
Inject Time	40 msec	40 msec	40 msec
Detector filament	on	on	on
Auto Zero	on	on	on
Detector Sensitivity	medium	medium	medium
Integrator/ Data System Conditions		Printer/Plotter chart speed	20 cm/min
		Printer/Plotter Attenuation	256X
		Input Signal	1 Volt

7.7.2 Calibration

The M2001 software used with the M200 GC uses Retention Time Indices (RTIs) based on a homologous series of compounds, in this case n-alkanes. A calibration run using three or more n-alkanes is run at the appropriate GC conditions. The RTIs are updated in the library as per M2001 manual. The RTIs for the entire library are not updated by correcting stored library values against the current "experimental" calibration run. It is important to bracket the entire library retention time with the three or more n-alkanes used in the calibration run.

A second calibration run using the target compounds of interest is then performed. This second calibration establishes the correct response factor which will be used to calculate the concentrations of the target compounds. This second calibration is required because the first calibration, using the n-alkanes, will establish the correct RTIs but will not yield the correct response factors for the target compounds. Therefore, two calibrations, the first to establish the correct RTI, and the second to calculate the correct response factor, are required at least once a day. Note: the results for compounds not in the calibration run are estimated from the response factor of their nearest neighbor.

The generation of calibration standards can be performed either at the site or in the laboratory prior to entering the field. In the latter case, standards must still be run in the field to ensure that the calibration runs stored on the data disks are valid and close to standards run in the lab. Dilutions are typically made from the certified gas standards cylinders using Hamilton 500, 1000 and 1500 cm^3 model "Super syringes" and Tedlar sampling bags. Simple volumetric dilutions are made and the set of standards analyzed as if they were typical samples. If the sample pre-concentrator is used, the trapping efficiency must be recorded.

At least three concentrations of each standard must be run. Preferably more standards are analyzed to establish the minimum ranges for the linear

response of the detectors for each individual target compound. Linear regression must be performed and an R^2 value of 0.90 or greater should be achieved.

7.7.3 Sample Analysis

To analyze a sample, fill a 2.0 cm^3 glass syringe with the sample, attach a Teflon Luer lock to the syringe tip and carefully place this assembly into the sample port located on the unit. Make sure the Luer lock is in the open position. The internal sampling pump will automatically draw a portion of the sample from the syringe into the instrument, once the injection command is executed (Figure 3, Appendix D).

7.8 CALCULATIONS

A calibration curve of at least three concentrations must be constructed for each target compound. A straight line equation in the form of $y = (m)(x) + b$; *(where: x = concentration, y = area counts, m* = slope and b = the intercept) is fit to the standard's raw data. The (y), or the unknown concentration for the sample, is determined from the above straight line equation. Non-linear data is indicative of erroneous detector response. Alternatively, sample concentration can be calculated as below:

$$\text{Sample Conc.} = \frac{(\text{standard conc.})\ (\text{sample area})}{(\text{standard area})}$$

The M2001 software will automatically calculate the concentration based on the response factor generated in the calibration mode of the program. Calibrations can be single point or multipoint calculations.

7.9 QUALITY ASSURANCE/ QUALITY CONTROL

The following QA/QC protocols are applicable:

- A complete calibration curve must be run daily.
- Duplicates of a standard, in the mid-range of the calibration curve and preferably close to sample results, should be run every 10 samples to ensure constant detector response.
- Two or three duplicates for each sample should also be run. These duplicate responses should be within 10-20% of each other in terms of area count and retention time values.
- Matrix spikes, or spiking samples with known levels of standards, must be run along with the samples, and should bracket the levels found in the field samples.
- The same Tedlar bag may be analyzed by other field instrumentation (e.g., Photovac, OVA, Sentex, etc.) and/or collected into tubes for GC/MS confirmation. If Tedlar bags are used to prepare standards, the time of preparation should be noted.
- During sample analysis, one of the standards should be periodically re-analyzed to ascertain if any sample loss occurs in the bag over time.
- A performance evaluation sample (PE) is typically sent along to determine if any loss or contamination occurs from transit or handling during sampling.
- A trip blank of zero air is also sent and analyzed at the end of the sampling run to determine if any contamination of the Tedlar bags occurred during transit.

7.10 DATA VALIDATION

The Retention Time Index (RTI) is used for peak identifications. If peaks are eluting close to the target compounds, sample spikes using known levels of target compounds can be prepared to identify the absence/presence of these target compounds in the samples. Typically, only the RTI is needed to identify the peaks of interest. Quantification is determined from the linear calibration curve, and by solving for concentration (y) from the straight line equation. The coefficient of variation on the straight line equation should have an R^2 of 0.90 or greater. Confirmation of the identity of any particular target compound must be done by other analytical methods, typically GC/MS.

Alternatively, a statistical approach to data validation can be sought. Once the linear range is established, analyze an appropriate standard, either a low or midrange concentration, 10 or more times throughout the day. Determine the standard deviation of the mean (σ(N-1)) for the response of the standard selected. The statistical method detection limit, or MDL, is 3 times the standard deviation (3σ). The method quantitation limit, or MQL, is then 10 times the standard deviation (10σ). Results below the MDL are considered "nondetects" (ND). Results above the MDL but below the MQL are considered "detected," but below the quantitation limit and thus are ascribed a "J" value. This "J" value flags the data as questionable. Results above the MQL are considered statistically reliable data.

7.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety practices. Analysis should be performed in a well-ventilated room. The sample vent port on the back of the M200 should be equipped with either a carbon scrubber or a long Tygon tube to vent sample gases outside of the work area. All carrier gas cylinders must be securely bolted to a table or piece of heavy furniture. When liquid reagents are used to prepare standards, the work should be performed under a vented hood with the analyst wearing safety glasses and disposable protective gloves.

APPENDIX A

Ionization Potentials

Ionization Potentials

Some Atoms and Simple Molecules			
Atom/Molecule	IP (eV)	Molecule	IP (eV)
H	13.595	I_2	9.28
C	11.264	HF	15.77
N	14.54	HCl	12.74
O	13.614	HBr	11.62
Si	8.149	HI	10.38
S	10.357	SO_2	12.34
F	17.42	CO_2	13.79
Cl	13.01	COS	11.18
Br	11.84	CS_2	10.08
I	10.48	N_2O	12.90
H_2	15.426	NO_2	9.78
N_2	15.580	O_3	12.80
O_2	12.075	H_2O	12.59
CO	14.01	H_2S	10.46
CN	15.13	H_2Se	9.88
NO	9.25	H_2Te	9.14
CH	11.1	HCN	13.91
OH	13.18	C_2N_2	13.8
F_2	15.7	NH_3	10.15
Cl_2	11.48	CH_3	9.840
Br_2	10.55	CH_4	12.98

Alkyl Halides			
Molecule	IP (eV)	Molecule	IP (eV)
HCl	12.74	CH_2BrCl	10.77
Cl_2	11.48	$CHBr_2Cl$	10.59
CH_4	12.98	ethyl bromide	10.29
methyl chloride	11.28	1,1-dibromoethane	10.19
dichloromethane	11.35	1-bromo-2-chloroethane	10.63
trichloromethane	11.42	1-bromopropane	10.18
tetrachloromethane	11.47	2-bromopropane	10.075
ethyl chloride	10.98	1,3-dibromopropane	10.07
1,2-dichloroethane	11.12	1-bromobutane	10.13
1-chloropropane	10.82	2-bromobutane	9.98
2-chloropropane	10.78	1-bromo-2-methylpropane	10.09
1,2-dichloropropane	10.87	2-bromo-2-methylpropane	9.89
1,3-dichloropropane	10.85	1-bromopentane	10.10
1-chlorobutane	10.67	HI	10.38
2-chlorobutane	10.65	I_2	9.28
1-chloro-2-methylpropane	10.66	methyl iodide	9.54
2-chloro-2-methylpropane	10.61	diiodomethane	9.34
HBr	11.62	ethyl iodide	9.33
Br_2	10.55	1-iodopropane	9.26
methyl bromide	10.53	2-iodopropane	9.17
dibromomethane	10.49	1-iodobutane	9.21
tribromomethane	10.51	2-iodobutane	9.09

Ionization Potentials (continued)

Alkyl Halides (continued)		Paraffins and Cycloparaffins	
Molecule	IP (eV)	Molecule	IP (eV)
1-iodo-2-methylpropane	9.18	methane	12.98
2-iodo-2-methylpropane	9.02	ethane	11.65
1-iodopentane	9.19	propane	11.07
F_2	15.70	n-butane	10.63
HF	15.77	i-butane	10.57
$CFCl_3$ (Freon 11)	11.77	n-pentane	10.35
CF_2Cl_2 (Freon 12)	12.31	i-pentane	10.32
CF_3Cl (Freon 13)	12.91	2,2-dimethylpropane	10.35
$CHClF_2$ (Freon 22)	12.45	n-hexane	10.18
$CFBr_3$	10.67	2-methylpentane	10.12
CF_2Br_2	11.07	3-methylpentane	10.08
CH_3CF_2Cl (Genetron 101)	11.98	2,2-dimethylbutane	10.06
$CFCl_2CF_2Cl$	11.99	2,3-dimethylbutane	10.02
CF_3CCl_3 (Freon 113)	11.78	n-heptane	10.08
$CFHBrCH_2Br$	10.75	2,2,4-trimethylpentane	9.86
CF_2BrCH_2Br	10.83	cyclopropane	10.06
CF_3CH_2I	10.00	cyclopentane	10.53
n-C_3F_7I	10.36	cyclohexane	9.88
n-$C_3F_7CH_2Cl$	11.84	methylcyclohexane	9.85
n-$C_3F_7CH_2I$	9.96		

Ionization Potentials (continued)

Aliphatic Alcohol, Ether, Thiol, and Sulfides		Aliphatic Aldehydes and Ketones	
Molecule	IP (eV)	Molecule	IP (eV)
H_2O	12.59	CO_2	13.79
methyl alcohol	10.85	formaldehyde	10.87
ethyl alcohol	10.48	acetaldehyde	10.21
n-propyl alcohol	10.20	propionaldehyde	9.98
i-propyl alcohol	10.16	n-butyraldehyde	9.86
n-butyl alcohol	10.04	isobutyraldehyde	9.74
dimethyl ether	10.00	n-valeraldehyde	9.82
diethyl ether	9.53	isovaleraldehyde	9.71
n-propyl ether	9.27	acrolein	10.10
i-propyl ether	9.20	crotonaldehyde	9.73
H_2S	10.46	benzaldehyde	9.53
methanethiol	9.440	acetone	9.69
ethanethiol	9.285	methyl ethyl ketone	9.53
1-propanethiol	9.195	methyl n-propyl ketone	9.39
1-butanethiol	9.14	methyl i-propyl ketone	9.32
dimethyl sulfide	8.685	diethyl ketone	9.32
ethyl methyl sulfide	8.55	methyl n-butyl ketone	9.34
diethyl sulfide	8.430	methyl i-butyl ketone	9.30
di-n-propyl sulfide	8.30	3,3-dimethyl butanone	9.17
		2-heptanone	9.33
		cyclopentanone	9.26
		cyclohexanone	9.14
		2,3 butanedione	9.23
		2,4-pentanedione	8.87

Ionization Potentials (continued)

Aliphatic Acids and Esters		Aliphatic Amines and Amides	
Molecule	IP (eV)	Molecule	IP (eV)
CO_2	13.79	NH_3	10.15
formic acid	11.05	methyl amine	8.97
acetic acid	10.37	ethyl amine	8.86
propionic acid	10.24	n-propyl amine	8.78
n-butyric acid	10.16	i-propyl amine	8.72
isobutyric acid	10.02	n-butyl amine	8.71
n-valeric acid	10.12	i-butyl amine	8.70
methyl formate	10.815	s-butyl amine	8.70
ethyl formate	10.61	t-butyl amine	8.64
n-propyl formate	10.54	dimethyl amine	8.24
n-butyl formate	10.50	diethyl amine	8.01
isobutyl formate	10.46	di-n-propyl amine	7.84
methyl acetate	10.27	di-i-propyl amine	7.73
ethyl acetate	10.11	di-n-butyl amine	7.69
n-propyl acetate	10.04	trimethyl amine	7.82
isopropyl acetate	9.99	triethyl amine	7.50
n-butyl acetate	10.01	tri-n-propyl amine	7.23
isobutyl acetate	9.97	formamide	10.25
sec-butyl acetate	9.91	acetamide	9.77
methyl propionate	10.15	N-methyl acetamide	8.90
ethyl propionate	10.00	N,N-dimethyl formamide	9.12
methyl n-butyrate	10.07	N,N-dimethyl acetamide	8.81
methyl isobutyrate	9.98	N,N-diethyl formamide	8.89
		N,N-diethyl acetamide	8.60

Ionization Potentials (continued)

Other Aliphatic Molecules with N Atom		Olefins, Cyclo-olefins, Polenes, Acetylenes	
Molecule	IP (eV)	Molecule	IP (eV)
nitromethane	11.08	ethylene	10.515
nitroethane	10.88	propylene	9.73
1-nitropropane	10.81	1-butene	9.58
2-nitropropane	10.71	2-methylpropene	9.23
HCN	13.91	trans-2-butene	9.13
acetonitrile	12.22	cis-2-butene	9.13
propionitrile	11.84	1-pentene	9.50
n-butyronitrile	11.67	2-methyl-1-butene	9.12
acrylonitrile	10.91	3-methyl-1-butene	9.51
3-butene-nitrile	10.39	3-methyl-2-butene	8.67
ethyl nitrate	11.22	1-hexene	9.46
n-propyl nitrate	-----	1,3-butadiene	9.07
methyl thiocyanate	10.065	isoprene	8.845
ethyl thiocyanate	9.89	cyclopentene	9.01
methyl isothiocyanate	9.25	cyclohexene	8.945
ethyl isothiocyanate	9.14	4-methylcyclohexene	8.91
		4-cinylcyclohexene	8.93
		cyclo-octatetraene	7.99
		acetylene	11.41
		propyne	10.36
		1-butyne	10.18

Ionization Potentials (continued)

Some Derivatives of Olefins		Heterocyclic Molecules	
Molecule	IP (eV)	Molecule	IP (eV)
vinyl chloride	9.995	furan	8.89
cis-dichloroethylene	9.65	2-methyl furan	8.39
trans-dichloroethylene	9.66	2-furaldehyde	9.21
trichloroethylene	9.45	tetrahydrofuran	9.54
tetrachloroethylene	9.32	dihydropyran	8.34
vinyl bromide	9.80	tetrahydropyran	9.26
1,2-dibromoethylene	9.45	thiophene	8.860
tribromoethylene	9.27	2-chlorothiophene	8.68
3-chloropropene	10.04	2-bromothiophene	8.63
2,3-dichloropropene	9.82	pyrrole	8.20
1-bromopropene	9.30	pyridine	9.32
3-bromopropene	9.7	2-picoline	9.02
$CF_3CCl=CClCF_3$	10.36	3-picoline	9.04
$n\text{-}C_4F_{11}CF=CF_2$	10.48	4-picoline	9.04
acrolein	10.10	2,3-lutidine	8.85
crotonaldehyde	9.73	2,4-lutidine	8.85
mesityl oxide	9.08	2,6-lutidine	8.85
vinyl methyl ether	8.93		
allyl alcohol	9.67		
vinyl acetate	9.19		

Ionization Potentials (continued)

Aromatic Compounds			
Molecule	IP (eV)	Molecule	IP (eV)
benzene	9.245	2-methylnapthalene	7.955
toluene	8.82	biphenyl	8.27
ethyl benzene	8.76	phenol	8.50
n-propyl benzene	8.72	anisole	8.22
i-propyl benzene	8.69	phenetole	8.13
n-butyl benzene	8.69	benzaldehyde	9.53
s-butyl benzene	8.68	acetophenone	9.27
t-butyl benzene	8.68	benzenethiol	8.33
o-xylene	8.56	phenyl isocyanate	8.77
m-xylene	8.56	phenyl isothiocyanate	8.520
p-xylene	8.445	benzonitrile	9.705
mesitylene	8.40	nitrobenzene	9.92
durene	8.025	aniline	7.70
styrene	8.47	fluoro-benzene	9.195
α-methyl styrene	8.35	chloro-benzene	9.07
ethynylbenzene	8.815	bromo-benzene	8.98
napthalene	8.12	iodo-benzene	8.73
1-methylnapthalene	7.96	o-dichlorobenzene	9.07

Aromatic Compounds (continued)		Miscellaneous Molecules	
Molecule	IP (eV)	Molecule	IP (eV)
m-dichlorobenzene	9.12	ethylene oxide	10.565
p-dichlorobenzene	8.94	propylene oxide	10.22
1-chloro-2-fluorobenzene	9.155	p-dioxane	9.13
1-chloro-3-fluorobenzene	9.21	dimethoxymethane	10.00
1-bromo-4-fluorobenzene	8.99	diethoxymethane	9.70
o-fluorotoluene	8.915	1,1-dimethoxyethane	9.65
m-fluorotoluene	8.915	propiolactone	9.70
p-fluorotoluene	8.785	methyl disulfide	8.46
o-chlorotoluene	8.83	ethyl disulfide	8.27
m-chlorotoluene	8.83	diethyl sulfite	9.68
p-chlorotoluene	8.70	thiolacetic acid	10.00
o-bromotoluene	8.79	acetyl chloride	11.02
m-bromotoluene	8.81	acetyl bromide	10.55
p-bromotoluene	8.67	cyclo-$C_6H_{11}CF_3$	10.46
o-iodotoluene	8.62	$(n\text{-}C_3F_7)(CH_3)C=O$	10.58
m-iodotoluene	8.61	trichlorovinylsilane	10.79
p-iodotoluene	8.50	$(C_2F_5)_3N$	11.7
benzotrifluoride	9.68	isoprene	9.08
o-fluorophenol	8.66	phosgene	11.77

APPENDIX B

Photovac Maintenance and Calibration

Photovac Maintenance and Calibration: Supplement to SOPs #2107 and #2108

PHOTOVAC 10A10

Septum Change

The Photovac 10A10 uses a Teflon-faced, silicon rubber, 6-mm diameter septum. The Hamilton "Micro Sep" F-138 is suitable. The septum can easily be replaced with the following procedure.

1. Unscrew septum retainer.
2. Remove the old septum using the needle of one of the gas tight syringes available for sample injection.
3. Insert the new septum, Teflon face down.
4. Carefully screw the retainer back into place firmly, but without overtightening.

A 10 to 20 minute stabilization period may be required due to a temporary interruption of the carrier gas flow when the septum is changed.

Column Maintenance

The standard Photovac 10A10 is equipped with a 4 foot by 1/8-inch OD Teflon tube packed with 3% SE-30 on 80/100 mesh Chromosorb G for field surveys and analyses requiring detailed separations. Normally the column will be connected for manual operation.

New columns must be conditioned overnight with ultrahigh purity helium (or nitrogen) at a temperature of 100°C and a flow rate of 10 cm^3/min. Reconditioning of older columns is accomplished under the same conditions.

To access the column, utilize the following procedures:

1. Never remove the panel while the instrument is connected to the main power supply.

Table F: Photovac Maintenance and Calibration Schedule

Function	Procedure/Frequency
Battery Charge (when instrument has been operating exclusively on wall current)	Charge for 10 hours on "low" setting; every 3 months.
Battery Charge (when instrument has been operating exclusively on batteries)	Charge for 1 1/2 hours on "high" setting for every hour of use (don't overcharge); after each use.
Calibration	Calibrate after every 24-hour period of use (see section 4.7.2).
Septum Change	Change after approximately every 40 sample injections (see section 4.7.5).
Column Reconditioning	Recondition every 3 months, after heavy use, or whenever installing a new column (see section 4.7.6).

2. Disconnect the AC cord.

3. Disconnect the chart recorder lead.

4. Disconnect the lecture bottle carrier gas supply.

5. Remove the four Phillips screws securing the panel to the case and remove the screw attaching the lid.

6. Grasp the panel assembly by the cylinder clamp. Gently lift the rear of the panel clear of the case rim and ease the panel assembly backward from the front rim. Lift the panel assembly clear.

7. Gently remove the wire harness connection from the circuit board into which it is plugged. Remove the nine Phillips screws from the gold box and lift clear the lid/circuit board subassembly. The interior of the column/ion cell chamber is now accessible.

8. To remove the column, locate the 2 compression fittings at either end of the column (ion cell body and injection port). Using a 5/16-inch, open-ended wrench, loosen these fittings. Unscrew the fitting with your hand and remove column.

9. To replace the column, reverse the previous steps and take special care not to damage the threads on the compression fittings. Fittings are made finger tight and then the 5/16-inch open-ended wrench is used to give an additional 1/8-inch turn to assure fitting seating.

PHOTOVAC 10S SERIES

UV Source

The ultraviolet light source is available to check proper performance. Occasional starting problems can be encountered. After switching the Photovac on, the LCD will respond with "Lamp not ready please wait." This is superseded by "Ready enter command" as soon as the lamp ignites.

Septum

The septum is 6-mm in diameter and composed of silicone rubber with a Teflon face. The Teflon face is always mounted downwards in the injection port.

1. To change the septum, take a spare syringe needle and push it down the septum retainer channel, so that it penetrates the septum.

2. Leaving the needle in place, unscrew the retainer and withdraw it, with the septum still impaled on the needle. Handle the new septum as little as possible between your fingers.

3. Using the left hand, position the new septum, (Teflon face down) at the end of the retainer and push the needle through with the right hand to hold the septum in the correct alignment.

4. Carefully screw the retainer back into position. The needle will maintain the septum in the correct position until it is seated.

The septum retainer must not be over tightened, as this will cause unusual resistance to penetration by the needle and may cause needle blockage.

Column Maintenance

The 10S Series Photovac is usually supplied with a 6-inch 5% SE-30 pre-column and a 4-foot 5% SE-30 analytical column suitable for field screening a variety of chlorinated and nonchlorinated alkene and aromatic compounds with adequate resolution. The SE-30 (dimethylsilicone gum) is a packed column. An alternative column, the CP Sil-5 (Dimethylpolysiloxane) capillary column, is also suitable for general field screening analysis.

New packed columns and heavily contaminated columns must be conditioned/reconditioned prior to use, using the column conditioning adaptor and UHP helium or nitrogen, at a flow rate of 5-10 cm^3/min, at 100°C overnight.

The capillary column is conditioned overnight with the same carrier gas. However, the maximum recommended temperature is 50°C if using the plastic encased capillary column supplied by Photovac International.

To access the column, utilize the following procedure:

1. Release the catch of the computer module located at the upper left hand corner of the module.

2. Raise the hinged module and secure in the open position.

3. Carefully unscrew compression fittings and remove the pre-column and analytical column.

4. To replace the columns, tighten the Teflon compression fittings, finger tight, being careful not to strip the threads, as this may result in leakage.

When the Photovac is not in use, it is advisable to maintain a carrier gas flow (2-5 cm^3/min) through the column and Photovac valve system. This will reduce possible contamination from outside sources, and off gassing of possible contaminants which may have accumulated in the detector or injection port. This will also aid in readiness for emergency response operations.

Batteries

The 10S Photovac portable GC uses a single 12V, 6-A rechargeable lead/acid battery. It is usually sufficient for at least 8 hours of continuous operation, depending on the duty cycle chosen for the valves in the case of continuous monitoring. The battery is guaranteed for 300 charge/discharge cycles which is equivalent to a year of very heavy activity.

A battery declining in charge will make itself known to the user by automatically shutting off the instrument. It will require an overnight charge.

In order to re-charge, connect the unit to the AC main. As long as the instrument is connected to the main, the batteries will be trickle charged or in a standby mode. Battery charging and monitoring are matters which are under the control of the computer; to check status, depress the "up arrow" key. Full charge will show 13.6 volts. When charging overnight, leave unit on with carrier flow at 5-7 mL/mm. This is equivalent to a high charge.

Printer/Plotter Service

As a general rule, this device requires little service beyond the timely replacement of pens and paper.

To replace a pen, turn the Photovac on and depress the <PEN> key. The pen turret will move to the right and stop. If the correct color Pen is uppermost, engage your fingernail in the ridges on the little nylon tab, located in the bottom right hand corner of the printer aperture and pull the tab towards you to eject the rear-end of the pen.

If the turret presents the wrong color pen for replacement, briefly depress the <PEN> key and the turret will rotate 90 degrees and present the next color, and so on. Lift the pen out and insert a new one, making sure that the ball tip passes through the tiny aperture in the metal plate located close to the writing surface. Press the rear-end of the new pen into its position in the turret and briefly depress the <FEED> key to bring the printer back "on line."

Both paper and pens are readily available from any "Radio Shack" or similar store. In the event of any difficulty in locating a supply, contact Photovac. Troubleshooting information and corrective action procedures can be found in Appendix C.

APPENDIX C

Troubleshooting Guides

Troubleshooting Guide for Photovac 10A10 SOP #2107

	Problem	Probable Cause	Remedy
1	No chromatographic response.	No carrier gas flow.	Check at *Out* port with flow gauge.
		Batteries flat (if on battery operation).	Plug into AC and check again.
		Electrometer saturated.	Turn "attenuation" to set meter to 0, if "offset" reads 10 or more, the instrument is saturated.
2	Unacceptable baseline drift.	Unit has been subjected to large temperature change.	Allow to stabilize until clear.
		A very concentrated sample has recently been introduced, resulting in excessive "tailing".	Allow to self purge until clear.
		Unacceptable contamination levels in carrier gas supply.	Change carrier gas supply and allow instrument to stabilize.
		The unit is charging and the resulting heat is affecting the column.	Turn *Charge* switch to *Off*.
3	Deterioration of sensitivity.	Syringe has leaky plunger.	Try new syringe.
		Column needs conditioning.	Condition column.
		Septum leaking.	Change septum.
		Column fittings leak.	Disassemble and check for leaks around fittings with soap solution, while under pressure.
		Deterioration due to ozone contamination after 1-3 years of operation.	Decrease attenuation; replace detector.
4	Unacceptable low frequency noise.	Column needs conditioning.	Condition column.
5	Peaks elute very slowly.	Carrier flow rate is too slow.	Increase flow rate.
6	Peaks eluting too fast.	Carrier flow rate is too high.	Decrease flow rate.
7	Peak has flat top.	Electrometer has saturated.	Lower injection volume. Pre-dilute sample and repeat.

	Problem	Probable Cause	Remedy
8	Peak is misshapen with considerable tailing.	Improper injection.	Repeat.
		Flow is too slow.	Increase flow.
		Improper injection technique.	Repeat.
		Peak is developing from an earlier injection.	Allow greater time between injections or install shorter column.
		Compound is wrongly matched to Column.	Select appropriate column.
		Power supply inadequate.	
9	Source off light stays on after 5 minutes.	Batteries low (if battery operated).	Plug in AC connector.
			Adjust potentiometer under aluminum cylindrical case by 45° clockwise increments.
		Wire attachments to power supply not secure.	Secure wire attachments.
		Tube driver mismatched.	Contact Photovac for advice (516)351-5800.
		Power supply inadequate.	
10	Electrometer does not return to zero.	Electrometer saturated.	Allow carrier flow for extended period without sample injection.

Troubleshooting Guide for Photovac 10S: Supplement to SOP #2108

Lamp won't light

Note: if the lamp won't light, turn the instrument off. If the instrument is left on for more than 3 minutes, the oscillator board will be blown.

1. First try tuning the lamp using the Photovac **plastic screwdriver**, at the detector chamber box. Lamp source tuning and replacement procedures follow.

   a. The Photovac UV source is located inside the black box marked PHOTOVAC, which is situated beneath the hinged electronic module. There is access for a special tuning tool, through an identified hole at the rear of the lid. The tool is provided and is located in a special clip on the front face of the black box. **Do not use a metal tool for this adjustment** as this may cause an electrical short circuit.

   b. Observe the "Lamp not ready, please wait" message seen on the LCD. Engage the tool in the slot of the small screw beneath the hole in the box lid. Rotate the screw slowly in a clockwise direction. You may have to make several slow rotations until the message changes to "Ready enter command." Note the screw repeats its setting every 360° of rotation. Once the "Ready" message appears, turn the instrument off (by pressing <ENTER>) and then turn it on again. After a maximum of 3 minutes, the "Ready" message should return. If it does not, repeat the procedure. If unable to achieve the "Ready" state at all, it is probable that the lamp has failed. Proceed as follows:

   c. Turn the instrument off. Remove the four securing screws from the lid of the detector box. Inside you will see a white Teflon cylinder screwed into the right wall of the box. This cylinder is the lamp holder and has a silver wire wound around it in a spiral. There is a connecting wire attached to the free end of the lamp holder. Pull this gently out of its socket. Gently unscrew the lamp holder and withdraw it: you will see the lamp inside as you pull it away from the wall. Be careful not to drop the lamp. Take the new lamp, being very careful not to touch the window end with your fingers. Remove the old lamp and replace it with the new one. The O-ring on the lamp should be positioned about 5 mm from the end face of the window. Screw the holder back into place. **Make sure it goes in straight and is not cross-threaded.** Tighten until resistance from the O-ring is felt. Replace the connecting wire into its socket. Replace the lid and make sure all four screws are returned and are tight.

   d. The instrument is now ready to start; press <ON> and tune if necessary.

   e. Tuning also affects the amount of power consumed by the instrument (lamp). Press <TEST> and <ENTER> to obtain the status report. Check to ensure the value of "POWER" is in the 20 to 50 range (200 to 500 mA). If the power exceeds 50, the battery life will be shortened significantly. Try to aim for a reading of 35 to 40, consistent with a reliable starting.

   f. While you are tuning the lamp, you can also check the power consumption without printing the status report.

   g. Press <TEST> and type in "7853" and <ENTER>. The message "Lamp power" followed by a number, possibly "30," will show. Then you can tune the lamp accordingly.

2. If, after a few tries at tuning the lamp, it still won't light, turn off the instrument. Remove the detector chamber lid, remove the yellow wire from the *S* port on the oscillator board. Now turn on the instrument, and using the plastic screwdriver tune the lamp to its brightest violet color. Replace the yellow wire into the *S* port, and replace the detector chamber lid.

3. As a last resort, change the lamp. See procedure 1.c above.

No Flow – Valves are Open

1. Check the carrier gas cylinder and regulator to ensure all valves are open.

2. Check flowmeter and injection port. If the injection port is over-tightened, the flow will be cut off.

3. Check column fittings (should only be finger tight) and check the plumbing configuration. If the GC is set up for serial flow, then either the green or black carrier gas tubing, the tee port on the capillary column, and the clear tubing coming from the top right side of the column chamber will be closed off. With the backflush setup the black carrier gas tubing will be going into the first port in valve 3. The green or blue valves 4 & 5, and the tee port on the capillary column will be hooked up to the last port in valve 3 via clear tubing.

Low Sensitivity

1. Leak in system.

2. Plugged syringe.

3. Gain too low.

No Peaks Appear

1. Plugged syringe.

2. Leak in system/no flow.

3. Check events (*Test* key) and all parameters; press <CHART ON WITH SETUP>, then press <START/STOP> and <ENTER>.

Keyboard Jam

1. A key is jammed when it won't respond to any commands. If one key is jammed the whole keyboard is jammed. To fix, lift off key with needle-nose pliers and lift up the metal tab with the needle-nose pliers. Replace the key and press down on the key, a clicking noise should be heard. Usually the last key pressed is the key that is jammed, also check the most frequently used keys.

2. As a last resort, hit the red reset button underneath the green piece of cardboard near the detector chamber. Re-enter all parameters.

Instrument Shuts Itself Off After Printing the Message, "Batteries low, AC power required"

1. This can be avoided two ways; first use a power surge cord, and second allow the GCs to run off their internal batteries for four (4) hours then charge overnight prior to taking the instruments out in the field. This will keep the batteries from going into deep discharge.

2. If the instrument does this out in the field, then run the GC using the Photovac external battery. (Note: it must be recharged every six hours.)

Pens won't Print

1. Check to make sure the paper is fed properly.

2. Check the metal tables in front of the printer barrel; they may be bent. With needle-nose pliers or tweezers, push the metal tabs towards the keyboard (away from the pen tips).

3. Make sure the metal pen holder on the pen barrel is not bent. Use a paint brush to brush away any dust from the printer.

Negative Baseline

1. Regular pressure is too low. Pressure should be 20-30 psi.

2. Check for a leak in the system.

3. Compounds from last run are going past the detector which causes the baseline to go negative, or the column needs more purging. An immediate fix is to put in two septa.

"Valve Remains On" Indicated on LCD.

"On" time greater than "off" time. Check "list" or "event" status on LCD.

Sensitivity Too Low

1. Calibration is wrong. Check.
2. Syringe is leaking. Check.
3. Probable leak in system. Finger tighten all suspect fittings. Check column attachments and injection port. **Do not over tighten fittings.**
4. Gain setting is too low. Increase.
5. Valve timing is wrong. Check EVENTS, it is possible that the backflush is too fast -- see above. It is also possible that injection time is too short.
6. Lamp is failing. Replace with spare and try again.

Peak appears but is not recognized

1. Peak not calibrated. Do qualitative calibration; see Exercise (8) in User's Manual.
2. Increase subsequent calibration frequency.
3 Check peak in library for proper ID, RT, and plotter numbers.

Peaks appear too slowly

Flow rate is too low.

Peaks appear too quickly

Flow rate is too high.

Battery life too short

Lamp power is too high. To get a status report, press <TEST> + <ENTER>. If "source power" is greater than 50, see Corrective Procedure 1 in Users Manual.

Printer/valve cycle is too frequent

If cycle time is 2 minutes you are using frequent full printout; battery life is reduced. Use external battery pack (Cat. No. SA202) or minimize print format.

Instrument uses carrier too fast

1. Flow rate is too high. Check flow rate.
2. Carrier gas is leaking. Tighten all fittings (especially on column). **Do not over tighten valve fittings.**

Troubleshooting Guide for the Micromonitor: Supplement to SOP #2111

If the ready asterisk (*) won't come on, perform the following:

1. Press the <METHOD> button. Note the temperature setpoint for column A. Press the <A/B> button. Note the temperature setpoint for column B.

2. Press the <STATUS> button. The display should show the status at the same value as the setpoint for B.

3. Press the <A/B> button. The display should show the status at the same value as the setpoint for A.

4. Press the <ENTER> button. The display should show a reading between 5 and 45 psig.

5. Press the <A/B> button. The display should show a reading in the same range of between 5 and 45 psig.

6. Press the <ENTER> button. The display should show a reading between +420 mV and -445 mV for the autozero voltage.

7. Press the <A/B> button. The display should still read in the above range.

8. If the temperatures are not correct, try waiting until they heat up or cool down.

9. If the pressures are not correct, check the supply or change the setting at the back.

10. If the autozero range is not correct, you may have a slowly eluting peak coming through the system.

11. Try increasing the temperature for a while to purge the column.

APPENDIX D

Figures

Figure 1: Pneumatics of Photovac 10S Series

SOP #2108

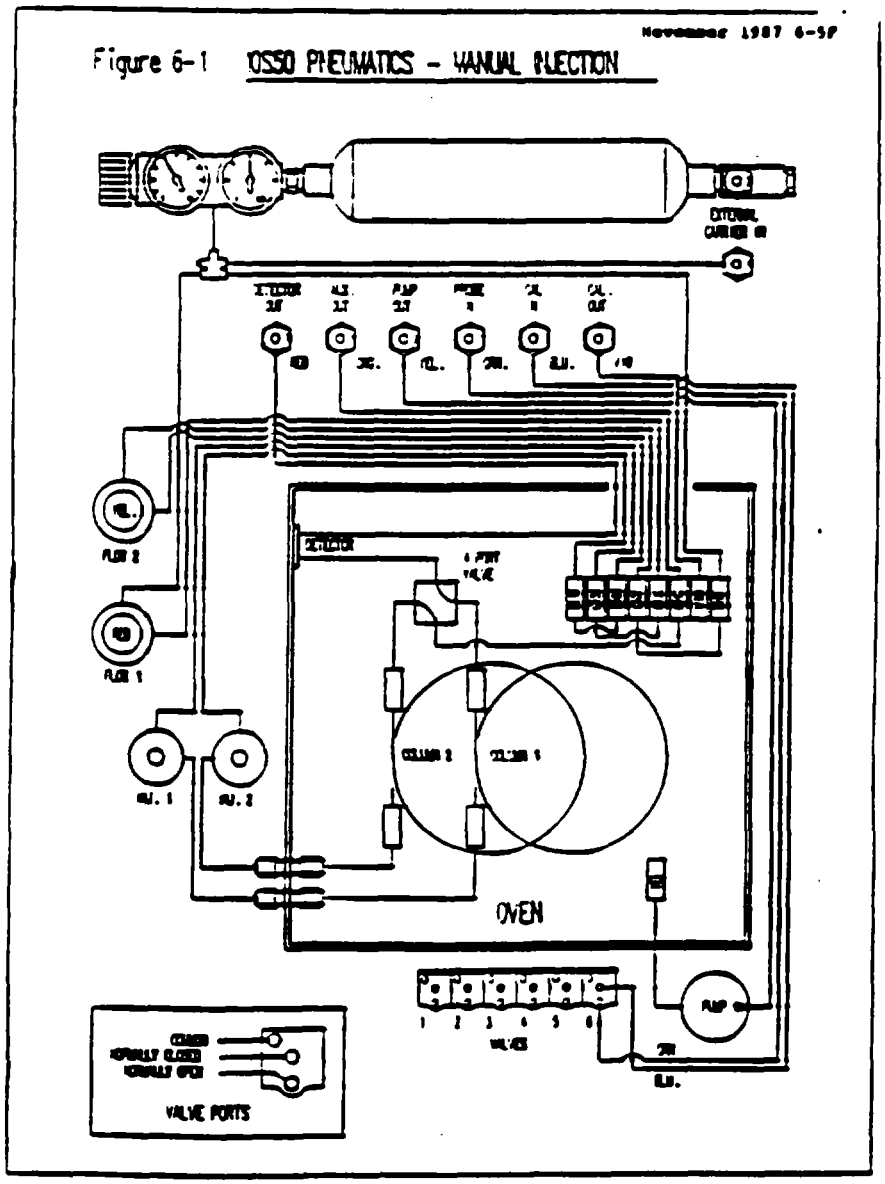


Figure 2: Micromonitor M200 Front Panel

SOP #2111

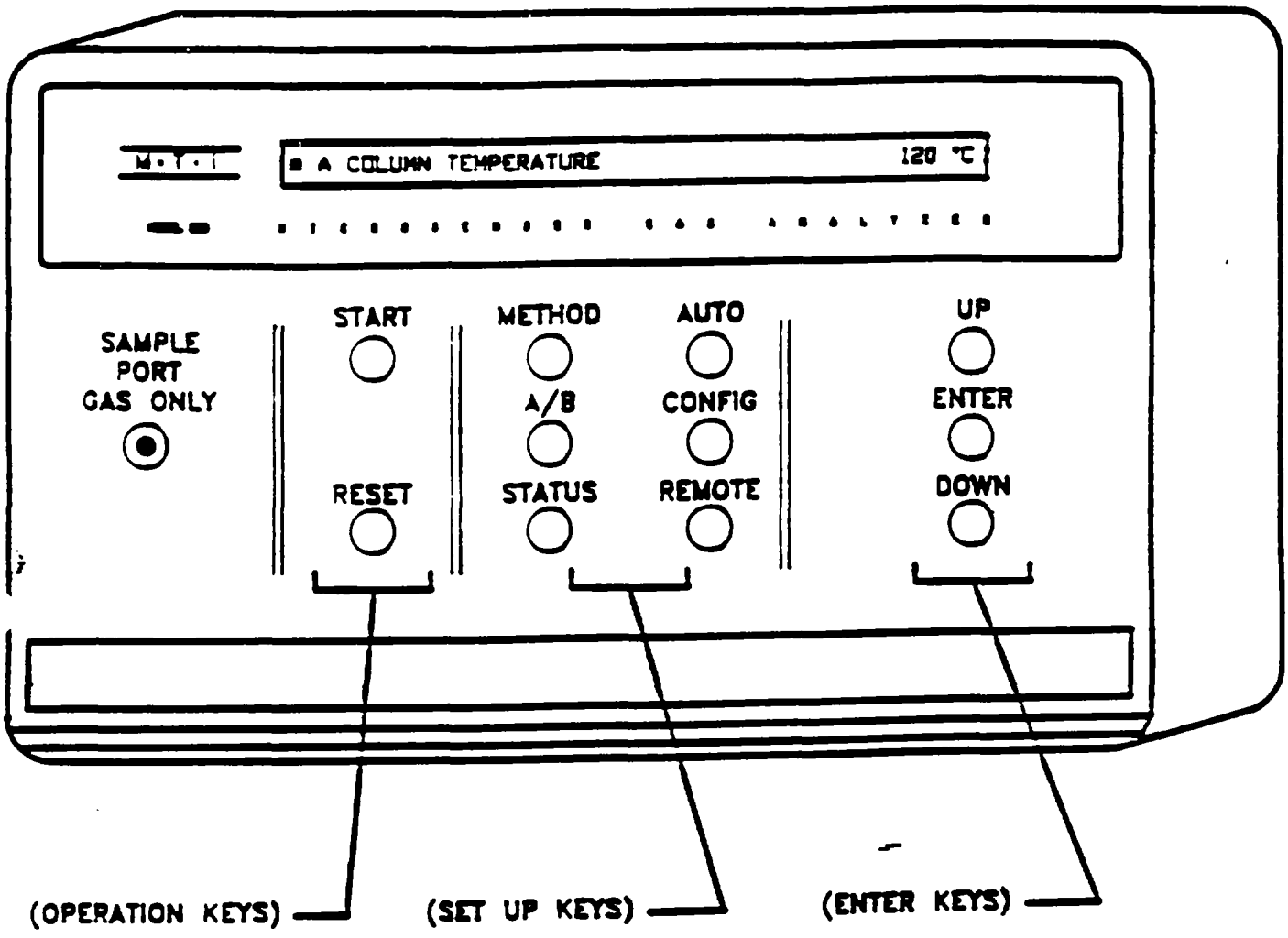


Figure 3: Systems Setup Diagram

SOP #2111

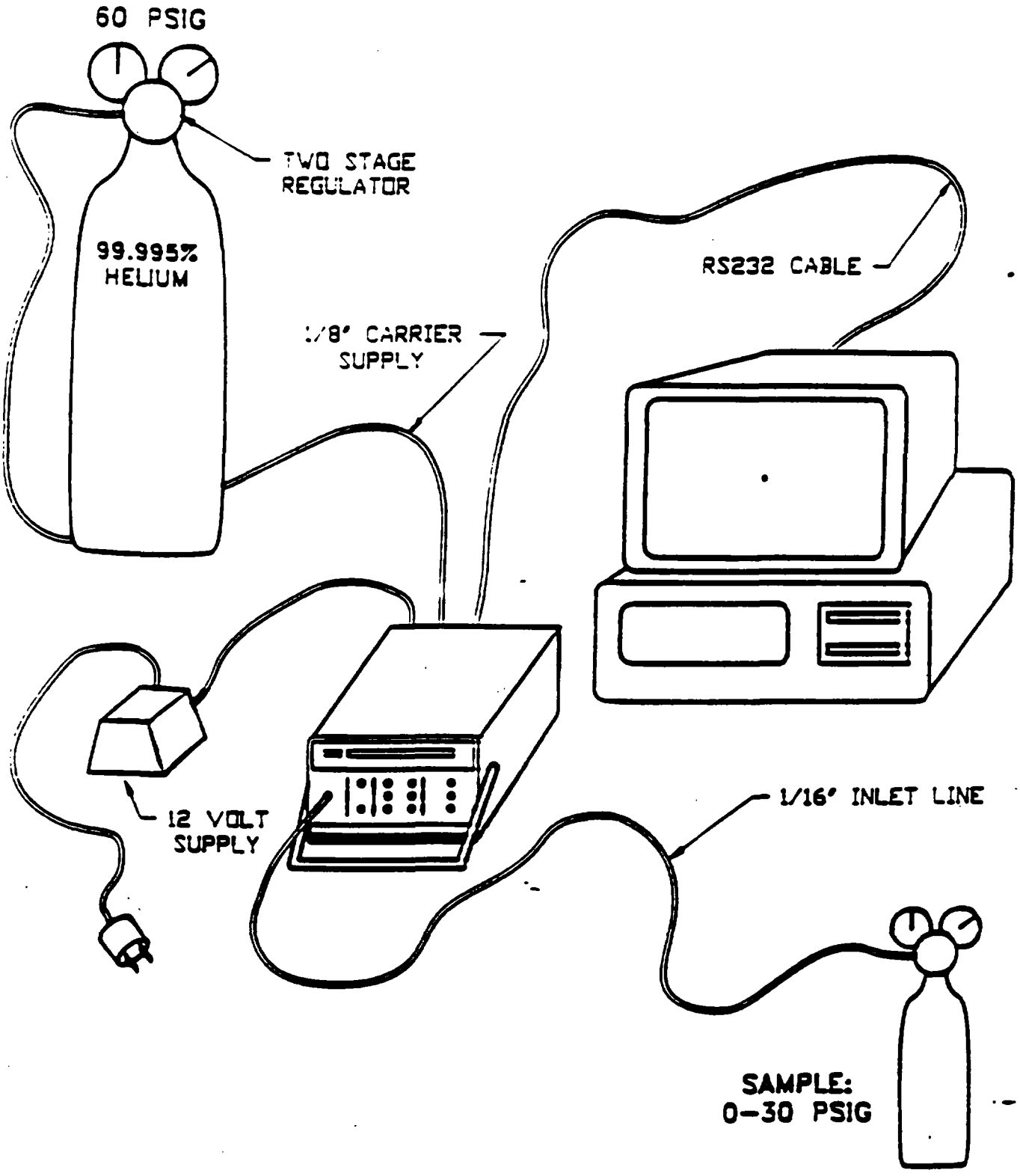
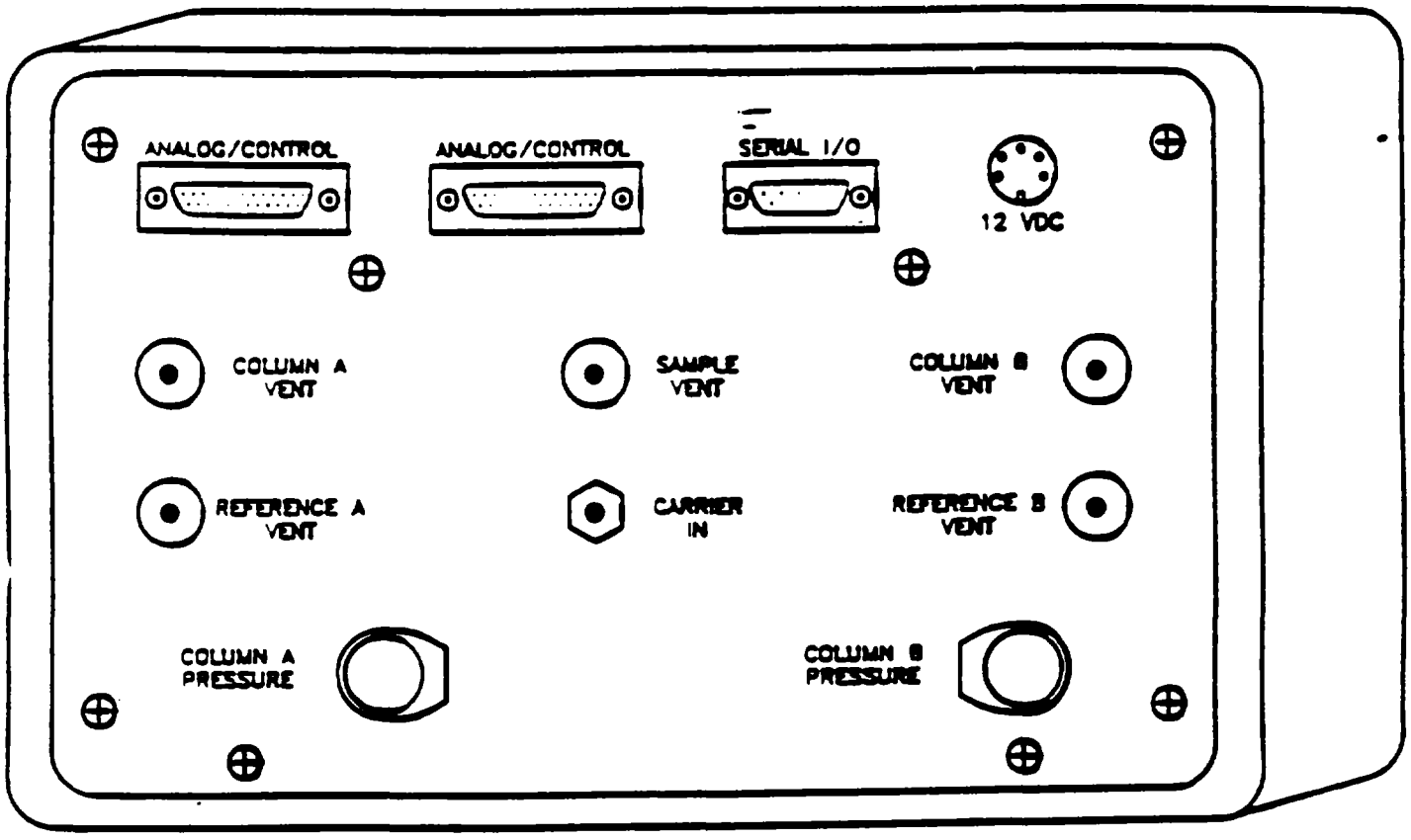


Figure 4: Micromonitor M200 Back Panel

SOP #2111



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clean atmospheres where targeted fiber concentrations are much less than 0.1 fibers/cm^3, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If more than 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L *in order to obtain an analytical sensitivity of 0.005* structures/cm^3. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25-mm (385 mm^2) and a 37-mm (855 mm^2) filter differ.

6.9 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements listed in the analytical method.

Generally field blanks are required for each set of samples or 10% of the total samples, whichever is greater.

The laboratory analyzing the samples should determine the lot blank requirements. There should be no less than one lot blank per cassette lot. It is preferable to have the lot blank analyzed prior to sampling.

6.9.1 Electronic Calibration -- Personal Sampling Pump

1. See the manufacturer's manual for operational instructions.

2. Set up the calibration train (as shown in figure 16, appendix A) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approximately 60 cm or 2 feet) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. *Perform the calibration three times until the* desired flow rate of ±5% is attained.

6.9.2 Electronic Calibration -- Rotameter

1. See the manufacturer's manual for operational instructions.

Table 9: Asbestos Sampling Flow Rates

	Concentration	Flow Rate
Low real-time monitor readings	< 6.0 mg/m^3	11 - 15 L/min
Medium real-time monitor readings	> 6.0 mg/m^3	7.5 L/min
High real-time monitor readings	> 10.0 mg/m^3	2.5 L/min

2. Set up the calibration train (as shown in figure 17, appendix A) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° of the vertical position.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range.

8. Perform the calibration three times until the desired flow rate of ±5% is attained.

Once on site, a secondary calibrator, such as a rotameter, may be used to calibrate sampling pumps.

6.9.3 Sampling Pump Calibration – Rotameter

1. See the manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (figure 18, appendix A) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approximately 60 cm or 2 feet) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° of the vertical position.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on site in the clean zone immediately prior to the sampling.

6.10 DATA VALIDATION

PCM analysis does not distinguish between asbestos and non-asbestos fibers; all fibers meeting the criteria are counted. TEM analysis can distinguish asbestos from non-asbestos fibers. This method of analysis should be used when the total fiber count is above the action level (or level of concern) so as to determine whether the airborne fiber is of asbestos origin.

Note: The flow rate and time should be adjusted to obtain optimum fiber loading on the filter.

6.11 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and site-specific health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

7.0 TEDLAR BAG SAMPLING: SOP #2050

7.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to define the use of Tedlar bags in collecting gaseous samples. Tedlar bags are used to collect both volatile and semi-volatile organic compounds, including halogenated and non-halogenated species. The sensitivity of the method is primarily instrument dependent.

7.2 METHOD SUMMARY

When collecting gaseous samples for analysis, it is often necessary to obtain a representative grab sample of the medium in question. The Tedlar bag collection system (see figure 19 in appendix A) allows for this and consists of the following items.

- Tedlar bag complete with necessary fittings
- desiccator in which the vacuum is created
- sampling pump to create the necessary vacuum
- appropriate Teflon and Tygon tubing

The Tedlar bag is placed into the desiccator and the fitting is inserted into Teflon tubing. The Teflon tubing is the path through which the gaseous medium will travel. The pump is attached to the Tygon tubing, which is part of the vacuum fitting on the desiccator. The pump evacuates the air in the desiccator, creating a pressure differential causing the sample to be drawn into the bag. The sample introduced into the Tedlar bag never passes through the pump. The flow rate for the pump must be defined prior to sampling (usually 3 L/min for bag sampling).

7.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The Tedlar bags most commonly used for sampling have a 1-liter volume, and are held in boxes of ten. When the sampling procedure is concluded, the Tedlar bags are stored in either a clean cooler or a trash bag to prevent photodegradation. It is essential that samples be analyzed within 48 hours, as after that time compounds may escape or become altered.

7.4 INTERFERENCES AND POTENTIAL PROBLEMS

Contamination is a major concern since many of the compounds in question will be present in the parts per billion range. The following practices will minimize the risk of cross-contamination.

- During transportation and storage, the further away from the source(s) of potential contamination the bags are, the less likely are the chances for external contamination.
- Bags must be attached only to clean Teflon tubing.
- Once the sample has been collected, affix the sample label to the edge of the bag to prevent adhesives on the label from permeating the body of the bag. Fill out labels with a ballpoint pen or a pencil, since permanent markers contain volatile compounds that may contaminate the sample.
- The chemical structure of Tedlar will cause highly polar compounds to adhere to the inner surface of the bag. Also, low molecular weight compounds may permeate the bag. Use real-time monitors such as the OVA, HNU, and CGI as a screening device prior to sampling. Write this information on the sample label to inform the individuals performing the sample analysis.

The Tedlar bag sampling system is straightforward and easy to use. However, be aware of the following when sampling.

- Ensure that the seal between the top half and the bottom half of the desiccator is air tight in order for the system to work.
- Check the O-ring gasket to see if it is in

place with the proper fit. O-rings that have been stretched out will not remain in place, requiring constant realignment.

- Check that all the fittings associated with the vacuum joints are securely in place. The fittings can be pushed loose when inserting the valve stem into the Teflon tubing.

- Check to ensure that a corner of the Tedlar bag is not jutting out between the two halves of the desiccator, thus impairing the seal.

- Be sure not to overinflate the bags. Overinflation will cause the bags to burst.

7.5 EQUIPMENT/APPARATUS

- Pelican cases, or desiccators -- cleaned, with Teflon tubing replaced, and equipped with extra O-rings.

- pump(s) -- charged, in good working order, and set with the appropriate flow rate of 3-L per minute.

- Tedlar bags -- free of visible contamination and preferably new.

7.6 REAGENTS

This section is not applicable to this SOP.

7.7 PROCEDURES

7.7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are needed.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or preclean equipment, and ensure that it is in working order.

4. Prepare a schedule. Coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.7.2 Field Operation

Tedlar bags are stored in boxes of 10. The valve is in the open position when stored. Occasionally, a piece of debris will clog the valve, necessitating the closing of the valve stem for it to clear. The valve stem is closed by pulling the stem out. If the valve stem is difficult to pull, it helps to twist the valve stem simultaneously.

1. Remove the Tedlar bag from the carton.

2. Insert the valve stem into the Teflon tube which runs through the desiccator.

3. Seal the desiccator by applying pressure to the top and bottom (ensure that the "O" ring is in place and unobstructed).

4. Connect the sampling pump to the evacuation tube.

5. Connect the intake tube to the desired source by placing the intake tube into the medium of concern.

6. Turn on the sampling pump.

7. Allow the bag to fill (indicated by the look of the bag as well as by the sound of the laboring pump).

8. Turn off the sampling pump and remove the evacuation tube from the pump.

9. Remove bag and pull the valve stem out.

10. Lock the valve stem.

11. Label the bag using either a tag or a sticker. Do not write on the bag itself.

12. Place Tedlar bag in a clean cooler or opaque trash bag to prevent photodegradation.

7.7.3 Post Operation

1. Once the samples are collected, transfer bags to the laboratory for analysis.

2. When transferring the Tedlar bags, a chain-of-custody form must accompany the samples. Personnel should be aware that some of the compounds of concern will degrade within a few hours of sampling.

3. Samples shipped must be in a clean cooler with a trip blank (a Tedlar bag filled with zero air) and a copy of the chain-of-custody form.

7.8 CALCULATIONS

This section is not applicable to this SOP.

7.9 QUALITY ASSURANCE/ QUALITY CONTROL

Depending upon the Quality Assurance Work Plan (QAWP) requirements, collect background samples consisting of upgradient/downgradient samples, or beginning/end of day samples, or a combination of the two. It may also be desirable to change sample train tubing between sample locations. Tedlar bag standards must be filled on site to identify the contaminants' degradation from the time the sample is collected until analysis. Tedlar bags filled with zero air must also accompany the sample bags to identify possible contamination during shipment and handling.

7.10 DATA VALIDATION

Results of the quality control samples (field and lot blanks) will be evaluated for contamination. This information will be utilized to qualify the environmental sample results according to the projects' data quality objectives.

7.11 HEALTH AND SAFETY

When working with potentially hazardous materials follow U.S. EPA, OSHA, and site-specific health and safety procedures.

8.0 CHARCOAL TUBE SAMPLING: SOP #2051

8.1 SCOPE AND APPLICATION

Charcoal tube sampling is utilized to identify specific contaminants in ambient air. The greatest selectivity of charcoal (activated carbon) is towards non-polar, organic, solvent vapors, (e.g., carbon tetrachloride, chlorobenzene and toluene). Organic compounds that are gaseous at room temperature, reactive, polar, or oxygenated (aldehyde alcohols and some ketones) are either not adsorbed (relatively early breakthrough), or inefficiently desorbed.

8.2 METHOD SUMMARY

Charcoal tube sampling is performed by drawing a known volume of air through a charcoal adsorption tube. As air is drawn through the tube, gases and vapors adsorb onto the surface of the charcoal. After sampling, the tubes are delivered to the laboratory for analysis.

8.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Charcoal used for sampling is housed in a glass tube that has been flame sealed. Charcoal tubes most often used contain either 150 mg or 600 mg of charcoal. The smaller 150-mg tube is 7-cm long with a 6-mm ID and a 4-mm OD containing two sections of 20/40 mesh activated carbon separated by urethane foam. The adsorbing section contains 100 mg of charcoal, the backup section 50 mg of charcoal. The larger 600-mg tube is 11-cm long with a 8-mm ID and a 6-mm OD containing two sections of 20/40 mesh activated carbon separated by urethane foam. The adsorbing section contains 400 mg of charcoal, the backup section contains 200 mg of charcoal. The larger tube can provide greater sensitivity by using a greater volume of air.

To preserve and store samples:

1. Place plastic caps on the charcoal tube ends.

2. Place the sample in a whirl bag. If duplicate samples have been collected, place both tubes in one whirl bag.

3. Indicate all applicable information on the chain-of-custody form, (e.g., sample volume, ID#, location, date, and weather parameters).

4. If the sample tube must be stored for more than a week, refrigeration is recommended. Maximum recommended holding time is two weeks.

5. Provide the name(s) of the analytical methodology(ies) being requested with the sample to the lab.

8.4 INTERFERENCES AND POTENTIAL PROBLEMS

High temperature and humidity, and high sampling flow rates may cause a decrease in the adsorption capacity of activated carbon. Contaminants from the front portion of the tube may migrate to the back portion of the tube. Refrigeration may minimize this migration.

8.5 EQUIPMENT/APPARATUS

8.5.1 Equipment List

- personal sampling pump
- dowel rods
- single or dual rotameter with stand and desired precalibrated flow rate
- charcoal tubes (600 mg or 150 mg)
- flexible PVC tubing (for attaching the tube holder system to the suction side of the pump)
- universal tube holder system
- sleeves (or support tubes to hold tubes in place)
- single or dual manifold flow controller
- tube holder end (tube holder ends support and seal the sampling tube within the plastic housing)
- glass cracker
- Ziploc bag
- whirl bags

- plastic caps

8.5.2 Equipment Source

While there may be other sources, tubes are readily available from SKC, Inc., and from Mine Safety Appliance Co., both of Pittsburgh, PA.

SKC: 1-800-752-8472
Mine Safety Appliance Co.: 1-800-MSA-2222

8.6 REAGENTS

This procedure utilizes totally dedicated equipment and does not require reagents.

8.7 PROCEDURES

8.7.1 Calibration

To save time in the field, sampling pumps can be precalibrated in the office prior to arriving at the site. The calibration must be checked in the field before and after sampling.

Assemble the calibration train as shown in figure 20 (appendix A), using a rotameter, sampling pump, manifold (low flow only) and representative charcoal tube. Use the same lot number of charcoal tubes for both sampling and calibrating.

1. Depending on the flow rate, adjust the sampling pump to the low- or high-flow mode (high flow > 750 cm^3/min).

2. For low flow calibration, turn the flow adjust screw on the manifold until the float ball on the rotameter is aligned with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

3. Affix a sticker to the manifold and pump indicating flow rate and media.

4. Remove the representative charcoal tube from the sleeve. The pump and manifold are calibrated as a unit and should not be separated until the samples have been collected. If the charcoal tube is run straight without a manifold, the calibration is performed by adjusting the flow directly on the pump.

8.7.2 Field Operation

1. Mobilize to the clean zone and calibrate the pumps.

2. Mobilize to the sampling location.

3. Crack the charcoal tube ends using a glass cracker.

4. Insert the charcoal tube in the sleeve with arrow pointing in the direction of air flow. (The smaller section is used for a backup and is positioned nearest the sampling pump.)

5. Screw the tip onto the sleeve so the charcoal tube is held in place.

6. Attach the sleeve(s) to a single or double manifold. At higher flow rates (>750 cm^3/min), charcoal tubes can run without a manifold. See figure 21.

7. To set up the sampling train, attach one end of the Tygon tubing (approximately 2 feet) to the tip of the sleeve or manifold. Attach the other end of the tubing to the inlet plug on the pump, figure 23 (appendix A).

8. Adjust time on the pump by adjusting past the zero mark several times to erase the pre-programmed time.

9. Place the charcoal tube in a vertical position on a dowel rod.

10. Record weather data (e.g., ambient temperature, barometric pressure, relative humidity, and wind direction).

11. Turn the pump on.

12. After the pump has run the full time, check the fault button to obtain the sample time. (This will indicate whether the pump ran for the scheduled time.)

13. Verify calibration.

8.7.3 Post Operation

1. Record the sampling time.

2. Remove the charcoal tube from the sleeve.

3. Immediately cap charcoal tubes with plastic caps. Never use rubber caps.

4. Place a sample ID# label on the tube.

5. Place the sample in a whirl bag labeled with the sample ID#, total volume, and required analysis. If duplicate samples have been collected, place both tubes in one whirl bag.

6. Indicate all applicable information on the chain-of-custody form (e.g., sample volume, ID#, location, date, and weather parameters).

7. If the sample tube must be stored for more than a week, refrigeration is recommended.

8. Provide the name(s) of the analytical methodology(ies) being requested to the lab with the samples.

To analyze the charcoal tubes, NIOSH Methods 1501, Aromatic Hydrocarbons; 1500, Hydrocarbons BP 36°-126°C; and 1003, Halogenated Hydrocarbons may be used. Other analytical parameters may be required. Determine the appropriate analytical methodology prior to field activities.

8.8 CALCULATIONS

The total volume of a sample is calculated by multiplying the total sample time by the flow rate. The total volume for each sample should be indicated on the chain-of-custody form.

8.9 QUALITY ASSURANCE/ QUALITY CONTROL

- Provide one field blank per sampling period or two field blanks for every 10 samples, whichever is greater. The tube should be handled in the same manner as the sampling tube (break, seal, and transport) except that no air is sampled through this tube.

- Provide a minimum of one appropriately labeled lot blank tube per sampling episode. The lab analyzing the samples can better determine the number of lot blank tubes required. These tubes are taken directly from the charcoal tube box. Do not break the ends.

- Provide one duplicate sample per 10 samples.

8.10 DATA VALIDATION

Results of the quality control samples will be evaluated. Utilize this information to qualify the environmental sample results in accordance with data quality objectives.

8.11 HEALTH AND SAFETY

Prior to initiating survey activities, a risk analysis is required to determine the hazards posed to sampling personnel. This will estimate any potential exposures to personnel, and define the extent of safety planning needed to complete the task.

Depending upon the hazards identified, a safety plan may be required prior to performing any site entry. In addition, real time monitoring may be necessary in order to verify ambient conditions and to determine adequate respiratory protection.

Specific hazards unique to charcoal tube sampling include:

- Sharp edges of the cracked tubes.

- Slip, trip and fall hazards at sampling locations.

9.0 TENAX TUBE SAMPLING: SOP #2052

9.1 SCOPE AND APPLICATION

Tenax/carbonized molecular sieve (CMS) tube sampling is utilized to identify specific contaminants in air. Compounds that can be determined by Tenax (U.S. EPA Method TO-1) are non-polar organics having boiling points in the range of approximately 80°C to 100°C. Compounds which can be determined by CMS are non-polar, non-reactive organics having boiling points in the range 15°C to 120°C. However, not all compounds falling into these category can be determined. Listed in table 10 below are many of the compounds which can be detected using Tenax/CMS. Analysis is performed by thermal desorption into a gas chromatograph/mass spectrometer/data system (GC/MS/DS).

9.2 METHOD SUMMARY

Tenax/CMS tube sampling is performed by drawing a known volume of air through a Tenax absorbent followed by a carbonized molecular sieve (CMS) adsorbent. Volatile organic compounds are captured on the adsorbent while major inorganic atmospheric constituents pass through or are only partially retained. After sampling, the tube is returned to the laboratory for analysis (U.S. EPA Method TO-1 and TO-2).

9.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Tenax/CMS tubes contain a granular inert chemical compound with adsorbent properties. A flame-sealed outer glass tube protects the Tenax/CMS tube from contamination. This outer glass tube must be broken prior to sampling. The Tenax/CMS air tube is 6-mm OD and 4-mm ID containing one section of 150 mg Tenax, 35/60 mesh and one section of 150 mg CMS 60/80 mesh.

Prior to site work, the culture tubes should be cleaned and prepared using the following procedure:

1. Place a plug of precleaned, silanized glass wool (methanol rinsed, baked in an oven at 120°C) in the bottom of each tube.
2. Place the culture tubes in an oven for at least 2 hours at 120°C. Do not bake the Teflon-lined caps.
3. Remove the culture tubes from the oven and allow them to cool.
4. Place the culture tubes in a Ziploc bag or whirl pack.

Table 10: Compounds Detected by Tenax/CMS

• benzene	• 1,2-dichloroethane	• toluene
• bromochloromethane[1]	• 1,1-dichloroethene	• 1,1,1-trichloroethane
• bromodichloromethane	• trans-1,2-dichloroethene	• 1,1,2-trichloroethane
• p-bromofluorobenzene	• ethylbenzene	• trichloroethylene
• carbon tetrachloride	• m-ethyltoluene	• trichlorofluoromethane
• chloroethane	• methylene chloride	• trichloromethane
• chloromethane	• styrene	• vinyl chloride
• dibromomethane	• 1,1,2,2,-tetrachloroethane	• m-xylene
• 1,1-dichloroethane	• tetrachloroethylene	• o-xylene

[1] Surrogate - Surrogates are injected into the Tenax tube to determine adsorption efficiencies.

Refrigerate the samples and keep out of sunlight. Storage for more than 4 weeks is not recommended.

9.4 INTERFERENCES AND POTENTIAL PROBLEMS

Contamination of the Tenax/CMS air tubes with the compound(s) of interest is a common problem. To minimize this problem, be extremely careful in preparing, storing, and handling the air tube throughout the sampling and analysis process. To avoid contamination from skin oils, use a lint-free glove when handling Tenax air tubes.

9.5 EQUIPMENT/APPARATUS

9.5.1 Equipment List

- calibrated personal sampling pump
- dual rotameter with stand and precalibrated flow rate
- Tenax/CMS tube, preferably of the same lot number
- flexible Tygon tubing (for attaching the tube holder system to the suction side of the pump)
- universal tube holder system
    - dual variable manifold flow controller
    - tube holder end with rubber boot adaptor
    - sleeves (clear plastic housings)
- glass cracker
- lint free cloth
- glass wool
- Teflon tape
- culture tubes

9.5.2 Equipment Sources

While there may be other sources, Tenax can readily be obtained from Supelco Inc., Bellefonte, PA, at (800) 247-6628; Technical Service (814) 359-3441 and MSA, 1-800-MSA-2222.

9.6 REAGENTS

Methanol is used in the lab to clean the glass tubing which holds the Tenax samples. Cleaning is performed prior to site work.

9.7 PROCEDURES

9.7.1 Calibration

1. Assemble the calibration train as shown in figure 23 using a rotameter, sampling pump, manifold, and representative Tenax tubes. Tenax tubes from the same lot are used for both sampling and calibration.
2. Adjust the sampling pump to the low-flow mode.
3. Remove the cap ends on the flow controller manifold. To adjust the flow, turn the needle valve with a small screwdriver (counter-clockwise to increase, clockwise to decrease).
4. Turn the flow-adjust screw on each manifold until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value (see figure 24).
5. Affix a sticker to the manifold and pump indicating the calibrated flow rate and media.
6. Remove the representative Tenax tubes from the sleeves.

The pump and manifold (including boots) are calibrated as a unit and should not be separated until the samples have been collected.

The pump and manifold are calibrated on-site in the clean zone immediately prior to sample collection. See table 11 for flow rate ranges and volumes.

Table 11: Recommended Flow Rates and Sample Volumes

	Flow Rate	Volume
Maximum	50 cm^3/min	5 liters
Optimum	30-40 cm^3/min	2 liters
Minimum	10 cm^3/min	0.5 liter

9.7.2 Field Operation

1. Calibrate pumps with manifolds as shown in section 9.7.1.

2. Crack the outer glass tube using a glass cracker.

3. Use a clean, lint-free cloth or gloves to remove the Tenax tube from the outer glass housing.

4. Insert the Tenax tube into a boot, with the carbonized molecular sieve section closest to the manifold.

5. Attach a protective sleeve over the tube. Do not enclose the Tenax tube end.

6. Set up the sampling train, by attaching one end of the Tygon tubing (approximately 60 cm or 2 feet) to the manifold; and the other end to the inlet plug on the pump (figure 25).

7. Place the sampling tube in the breathing zone. The pump and tube can be placed on a drum or hooked to a fence. A wooden dowel rod can also be used.

8. Position the tube either vertically or horizontally.

9. Adjust the pump time.

10. Turn the pump on.

11. Record weather data (e.g., ambient temperature, barometric pressure, relative humidity and wind direction).

12. Check the pump at the midpoint of the *sampling period if longer than 4 hours.*

9.7.3 Post Operation

1. At the end of the sampling period, check the fault button to obtain the run time. Record the run time. (This indicates whether or not the pump ran the full scheduled time.)

2. Check the flow rate and record the values in a field logbook.

3. Remove the Tenax tubes from sleeves using a lint-free cloth.

4. Place the Tenax tube in a culture tube. Tenax tubes from the same manifold and identical flow rates can be placed in the same culture tube.

5. Place a sample sticker indicating sample ID# on the culture tube. Do not put a sample sticker on the Tenax tube itself as this will contaminate the tube.

6. Attach the culture tube lid and wrap the lid/tube interface with Teflon tape.

7. Place the culture tubes into a Ziploc bag or a whirl pack.

8. Keep the samples refrigerated and out of sunlight. Storage for more than 4 weeks is not recommended.

9. Indicate all applicable information on the chain-of-custody form (e.g., sample volume, sample ID#).

10. Provide a copy of the air data sheets and the name of the preferred analytical methodology with the samples to the lab.

9.8 CALCULATIONS

The volume for each sample should be indicated on the chain-of-custody form.

Use the formula below to obtain the total volume:

Total Volume = Flow Rate × Time (minutes)

9.9 QUALITY ASSURANCE/ QUALITY CONTROL

Varying the sample volumes at the same location provides field QA/QC.

- Provide one appropriately labeled field blank per 10 samples. Handle this tube in the same manner as the sampling tube (break, seal, and transport), except that no air is sampled through this tube.

- Provide a minimum of one appropriately labeled lot blank tube per sampling episode. These tubes are taken directly

from the Tenax tube box. Do not break the outer glass housing. Place in a Ziploc bag and keep with other samples. Indicate the lot blank number on the chain-of-custody form.

- All sample stations should have duplicate sample tubes.

9.10 DATA VALIDATION

Results of the quality control samples (lot and trip blanks) will be evaluated for contamination. This information will be utilized to qualify the environmental sample results according to data quality objectives.

Data will be qualified according to acceptable variation on the prescribed flow rates (see table 11).

9.11 HEALTH AND SAFETY

Prior to initiating survey activities, an analysis of risk is required to determine the hazards posed to sampling personnel. This will estimate any potential exposures to personnel, and define the extent of safety planning needed to complete the task. Depending upon the hazards identified, a safety plan may be required prior to performing any site entry. In addition, real time monitoring may be necessary in order to verify ambient conditions and to determine adequate respiratory protection.

Specific hazards associated with Tenax tube sampling include:

- Small pieces of glass flying during "cracking" of the tube.

- Slip, trip and fall hazards at sampling locations.

10.0 POLYURETHANE FOAM SAMPLING: SOP #2069

10.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to outline the protocol for collection of polyurethane foam (PUF) samples. The PUF sampler is a complete air sampling system designed to simultaneously collect suspended airborne particulates and to trap airborne pesticide vapors. This system can efficiently collect a number of organochlorine and organophosphate compounds (e.g., dioxins, and polychlorinated biphenyls).

10.2 METHOD SUMMARY

Ambient air is drawn into a covered housing, then through a filter and foam plug by a high-flow-rate pump operating at a level of approximately 250 L/min (approximately 9 ft^3/min). This allows a sample of total suspended particulates (TSP) to collect on the filter surface. The foam plug allows collection of vapor which might be stripped from the particulates on the filter.

10.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Prior to sampling, ask the laboratory whether pre- and post-sampling filter weighing is appropriate.

After sampling, the foam plug and filter should be stored in an 8-oz. glass jar. The foam plug should occupy the bottom portion of the jar and the filter should be folded into quarters and placed on top of the plug. The jar is then wrapped with aluminum *foil (shiny side out).*

10.4 INTERFERENCES AND POTENTIAL PROBLEMS

Humidity can pose a problem; although glass fiber filters are comparatively insensitive to changes in relative humidity, collected particulate matter can be hygroscopic.

10.5 EQUIPMENT/APPARATUS

Specifications for equipment and supplies for monitoring ambient air for total suspended particulates (TSP) are provided in U.S. EPA's Reference Method: Determination of Suspended Particulates in the Atmosphere (High Volume Method) EPA/600/4-77/027a.

10.5.1 Sampling Media (Sorbents)

- polyurethane foam (PUF). Use polyether-type polyurethane foam (density No. 3014, 0.0225 grams/cm^3, or equivalent). This foam is the type generally used for furniture upholstery, pillows, and mattresses (General Metals Work's part number PSI-16 3-inch PUF plug is recommended, although 1- and 2-inch pieces are also available). This type of foam is white, and yellows on exposure to light. It should therefore be stored in a dark place (e.g., black trash bags or a cooler).

- 102-mm diameter glass fiber filter.

10.5.2 Sampling Equipment

- PSI PUF sampler or equivalent
- calibrated scale (if weighing is required)
- Teflon-coated tweezers
- aluminum foil
- hexane
- powder-free surgical gloves
- Solvex gloves
- sampling module holder
- plastic bag
- source of electricity (AC/DC): an electrical source of 100 volts, 15 amps is required

10.6 REAGENTS

Reagents are not used for preservation of PUF samples. Hexane is required for decontaminating PUF glassware. No other decontamination solutions are required.

10.7 PROCEDURES

10.7.1 Calibration of Timer, Meters and Standards

Elapsed-Time Meter

Every 6 months, the elapsed-time meter should be checked against a timepiece of known accuracy, either on site or in the laboratory. A gain or loss of more than 2 minutes per 24 hours warrants adjustment or replacement of the indicator. Record the results of these checks in the calibration logbook.

Flow Rate Transfer Standard

Calibration of the high-volume sampler's flow indicating device or the control device is necessary to establish traceability of the field measurement to a primary standard via a flow-rate transfer standard. The calibration procedures for orifice type flow transfer standards are listed in EPA's Test Method, 600/4-77/027a.

Upon receipt and at 1-year intervals, the calibration of the transfer standard orifices should be certified with a positive displacement standard volume meter (such as a Rootsmeter) traceable to the National Bureau of Standards (NBS). Calibration orifice units should be visually inspected for signs of damage before each use, and they should be recalibrated if the inspection reveals any nicks or dents in the orifice.

10.7.2 Field Calibration of High Volume Sampler

Calibration of the PUF sampler is performed without a foam plug and without filter paper in the sampling module. However, the empty glass cartridge must remain in the module to ensure a good seal through the module.

1. Connect the transfer standard orifice to the sample module. Ensure that no leaks exist between the orifice unit and the sampler.

2. Connect the orifice manometer to the orifice pressure tap.

3. Verify that the flow indicator is properly connected to the pressure tap on the lower side of the motor housing on the high volume sampler.

4. Set the manometer to "zero" as shown in figure 25 (appendix A).

5. Fully open the ball valve.

6. Fully open the voltage control screw. (Turn the screw next to the magnahelix gauge clockwise.)

7. Operate the sampler for at least 15 minutes to establish thermal equilibrium prior to calibration.

8. Adjust the voltage control screw to obtain the desired reading (perhaps 70) inches on the dial gauge (Magnahelix Gauge). A five-point calibration should be conducted in the range of the desired flow rate.

9. Record the dial gauge number 70 as your first calibration point, then read and record the pressure drop across the transfer standard orifice (H). Figure 25 (appendix A) demonstrates how to read the change in pressure drop.

10. Let the sampler run for at least 2 minutes to re-establish the run temperature conditions.

11. Adjust the voltage by moving the ball valve (red valve) to adjust the dial gauge down to 60 (arbitrary) inches. (Repeat steps 9-10.)

12. Using the above procedure (steps 9-11), adjust the ball valve for readings at 50, 40, and 30 inches.

13. Fully open the ball valve.

14. Turn the voltage-control screw clockwise as far as possible.

15. Measure and record the barometric pressure and ambient temperature on a field data sheet.

10.7.3 Sample Module Preparation

1. Put on powder-free surgical gloves.

2. Place the lower canister (figure 26, appendix A) sampling module in the module holder. All

sampling module in the module holder. All sampling equipment should be precleaned with hexane prior to use.

3. Check to ensure that the upper and lower orange silicone gaskets are in place (figure 26, appendix A).

4. Load the glass cartridge with a clean foam plug (with tweezers), making sure the foam is evenly distributed throughout the cartridge, and install in the module tube. (PUF plug should have been pre-cleaned with hexane by the laboratory that will be analyzing the samples.)

5. Install the filter holder assembly.

6. If filter weighing is required, weigh the 102-mm diameter glass fiber filter and record the weight in an analytical balance logbook. Calibrate an electronic balance; weighing paper filter is required.

7. Install lower Teflon gasket in the filter holder.

8. Handle the filter paper with Teflon-coated tweezers.

9. Place glass fiber filter (rough side up) into the filter holder.

10. Install the upper Teflon gasket.

11. Replace the 4-inch hold down ring and tighten the swing bolts.

12. Ensure that all fittings are snug, yet not overtight. (Overtightening will distort the gaskets.)

13. Cover the sample module with a clean plastic bag and place in a cooler.

14. Assemble a field blank and store in the same cooler.

It is recommended to have two sampling modules for each sampling system so that the filter and foam exchange can take place in the laboratory. The second set of modules is used for the subsequent sampling round.

10.7.4 Unit Operation

1. Transport the PUF sampler (figure 27, appendix A) to the desired location. The PUF sampler may be operated at ground level or elevated on scaffolding. The sampler should be located in an unobstructed area, at least two meters from any obstacle to air flow. In urban or congested areas, it is recommended that the sampler be placed on the roof of a single story building.

2. Calibrate the PUF sampler as indicated in section 10.7.2.

3. Adjust the exhaust hose downwind of the sampler.

4. Put on clean powder-free surgical gloves.

5. Place the loaded sampling module into the quick release fitting and engage by locking the two levers down securely.

6. Remove the plastic bag.

7. A field logbook or field data sheets should be used to record information (e.g., location, elapsed time meter, and time of day).

8. Turn the unit on.

9. Depending upon the desired flow rate, adjust the magnahelix gauge by turning the voltage control screw clockwise to increase, and counterclockwise to decrease the reading.

10. Wait approximately 2 minutes for the magnahelix gauge reading to stabilize, and then record it. The magnahelix dial gauge readings should be taken at the beginning and end of each sampling period. Differences between the two dial gauge numbers should be averaged.

11. Collect and average weather condition data during the sampling period, (e.g., wind direction, temperature, barometric pressure, and wind speed).

10.7.5 Unit Shutdown and Sample Collection

1. Using powder-free surgical gloves, open the shelter housing and record the magnahelix

gauge reading.

2. Turn the sampler off and record the elapsed time meter. Also, record the time of day.

3. Remove the sample module.

4. Cover the sample module with a polyethylene (plastic) bag. Keep the sample module in a vertical position at all times.

5. Place the sample module in a cooler. The field blank should also be stored in the same cooler.

6. Wearing Solvex gloves, wipe down the interior of the sampler with hexane and chem wipes.

7. If additional sampling is scheduled, install a new sampling module. The unit must be decontaminated with hexane and chem wipes prior to initiating another sampling round. If no additional sampling is scheduled, secure the unit.

8. Weigh the sample filter in a field laboratory, if required.

10.8 CALCULATIONS

Calculations are provided in U.S. EPA's Reference Method for Determination of Suspended Particulates in the Atmosphere (High-Volume Method), EPA/600/4-77/027a.

10.9 QUALITY ASSURANCE/ QUALITY CONTROL

Provide one field blank per sampling period or two field blanks for every 10 samples, whichever is greater.

10.10 DATA VALIDATION

Results of the quality control samples (field blanks) will be evaluated for contamination. This information will be utilized to qualify the environmental sample results in accordance with the data quality objectives.

10.11 HEALTH AND SAFETY

When working with potentially hazardous materials follow U.S. EPA, OSHA, and site-specific health and safety practices.

APPENDIX A

Figures

Figure 1: SUMMA Canister Cleaning System

SOP #1703

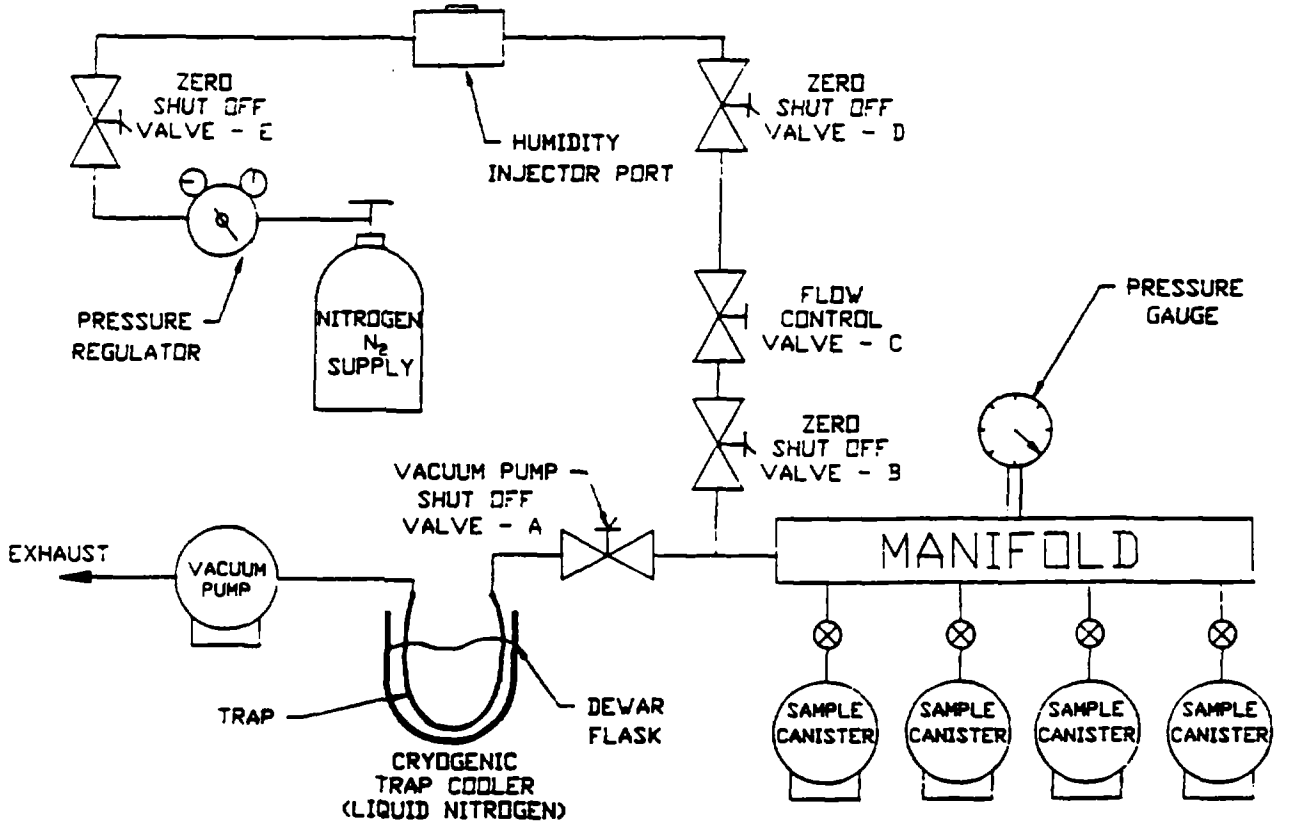


Figure 2: Pressurized and Subatmospheric Canister Sampling Systems

SOP #1704

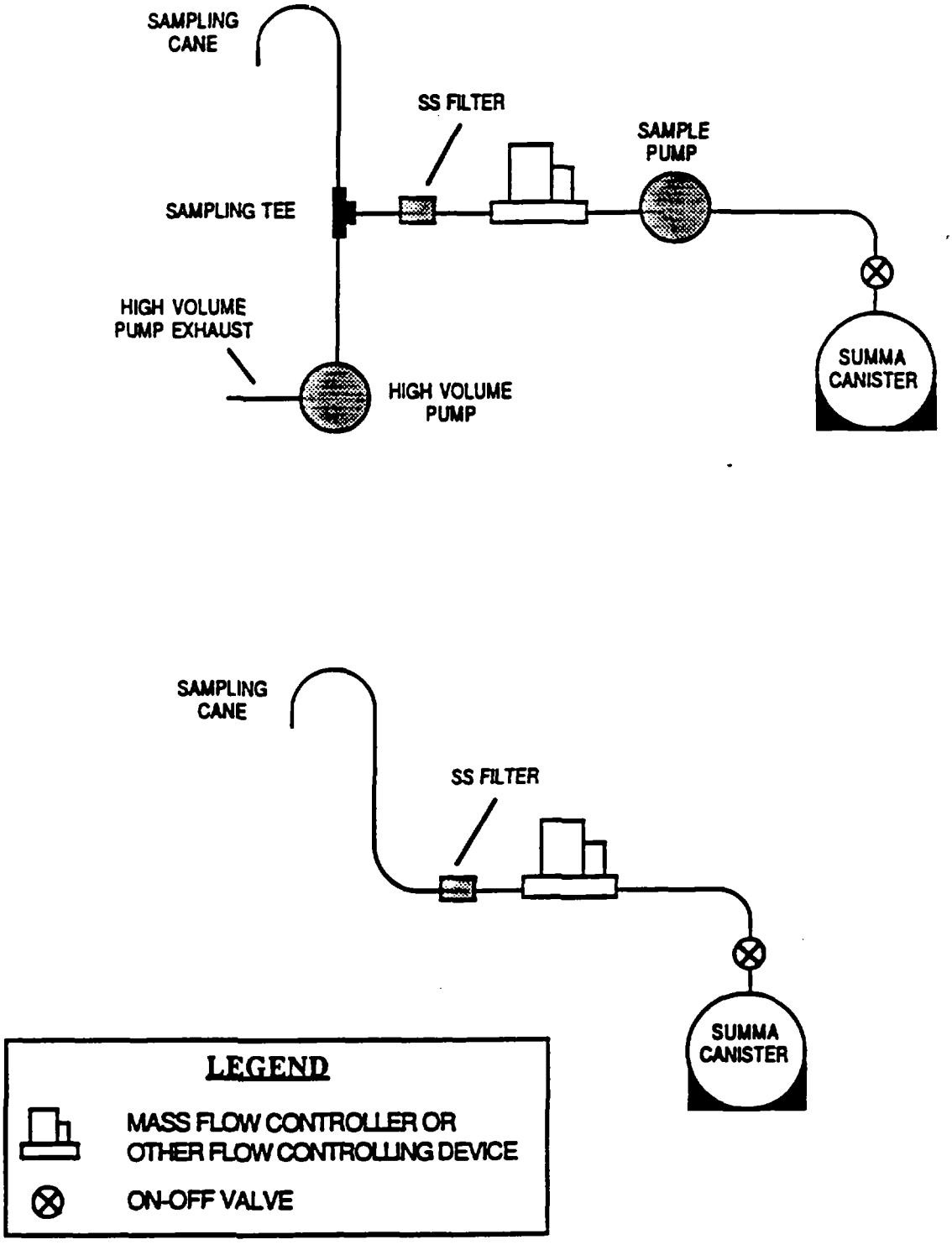


Figure 3: Tekmar Model 5010

SOP #1705

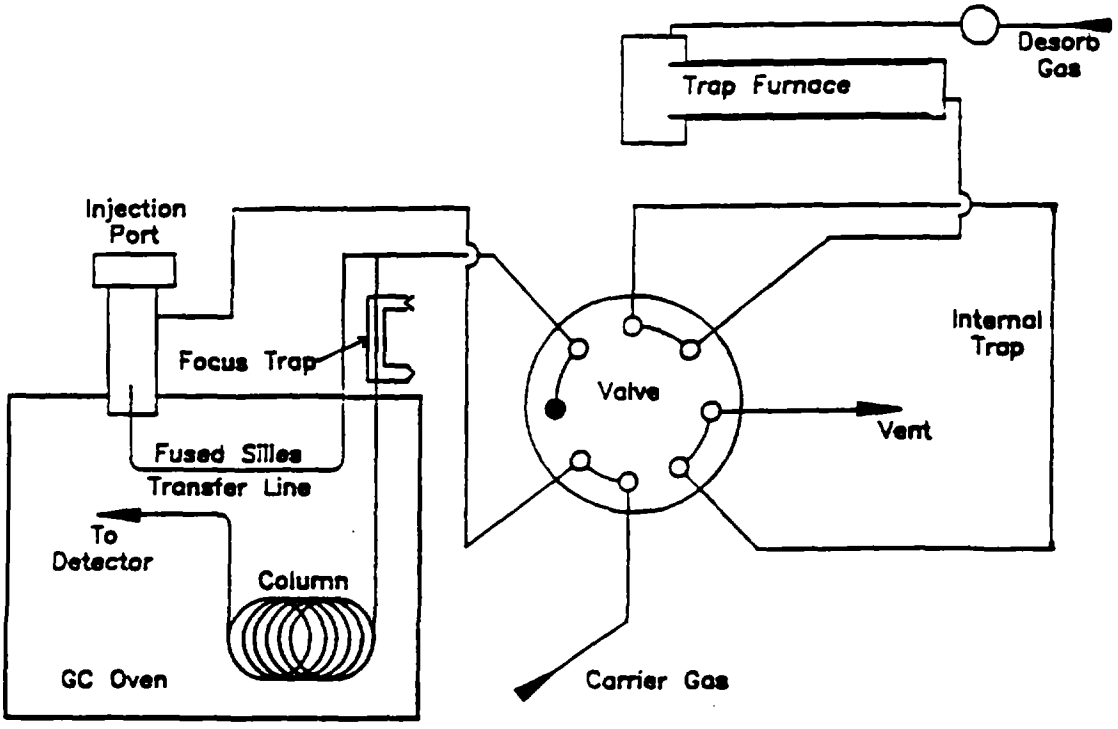


Figure 4: GC/MS Printout

SOP #1705

Operator ID: Bob
Output File: ^83874::D4
Data File: >83874::D4
Name: DAILY STANDARD
Misc: + 10 mL Surrogates

Quant Rev: 6

Quant Tme: 910416 14:30
Injected at: 910416 14:09
Dilution Factor: 1.00000

ID File: ID_SCT::D3
Title: GC/MS ANALYSIS OF TENAX/CMS CARTRIDGES (TO-1 & TO-2)
Last Calibration: 910411 14:17

#	Compound	R.T.	Scan #	Area	Conc.	Units	q
1)	#chloromethane	1.16	6	13555	664.87	PPR	74
2)	#vinyl chloride	1.25	16	13287	945.36	PPR	88
3)	#chloroethane	1.55	47	6583	881.16	PPR	93
4)	#trichlorofluoromethane	1.85	79	30141	814.42	PPR	95
5)	#1,1-dichloroethene	2.27	123	24379	825.44	PPR	88
6)	#methylene chloride	2.56	154	21909	803.94	PPR	93
7)	#trans-1,2-dichloroethene	3.15	216	25986	935.12	PPR	88
8)	#1,1-dichloroethane	3.50	253	29558	826.53	PPR	95
9)	#bromochloromethane	4.49	358	60788	1767.00	PPR	99
10)	#trichloromethane	4.55	364	35369	880.60	PPR	92
11)	#1,1,1-trichloroethane	5.24	432	32525	887.10	PPR	90
12)	#1,2-dichloroethane	5.39	453	28951	914.97	PPR	99
13)	carbon tetrachloride	5.67	482	25779	881.95	PPR	94
14)	#benzene	5.67	482	38009	775.10	PPR	93
15)	#trichloroethylene	6.77	598	23850	873.51	PPR	94
16)	#dibromomethane	6.79	601	29591	923.39	PPR	65
17)	#bromodichloromethane	6.98	620	35690	928.61	PPR	89
18)	#toluene	8.63	795	52178	888.48	PPR	87
19)	#1,1,2-trichloroethane	8.80	813	21806	892.69	PPR	89
20)	#tetrachloroethylene	9.76	914	34262	861.90	PPR	95
21)	#ethylbenzene	11.14	1060	72692	925.43	PPR	82
22)	#meta-xylene	11.34	1081	59722	939.36	PPR	92
23)	#styrene	11.88	1138	39679	1004.09	PPR	89
24)	#ortho-xylene	11.93	1143	64382	1008.13	PPR	79
25)	#1,1,2,2-tetrachloroethane	12.41	1194	53557	795.33	PPR	92
26)	#p-bromofluorobenzene	12.69	1223	37795	1142.98	PPR	98
27)	#meta-ethyltoluene	13.61	1320	21354	979.34	PPR	93

Compound uses FSTD

Figure 5: SUMMA Canister Sample Dilution Line

SOP #1705

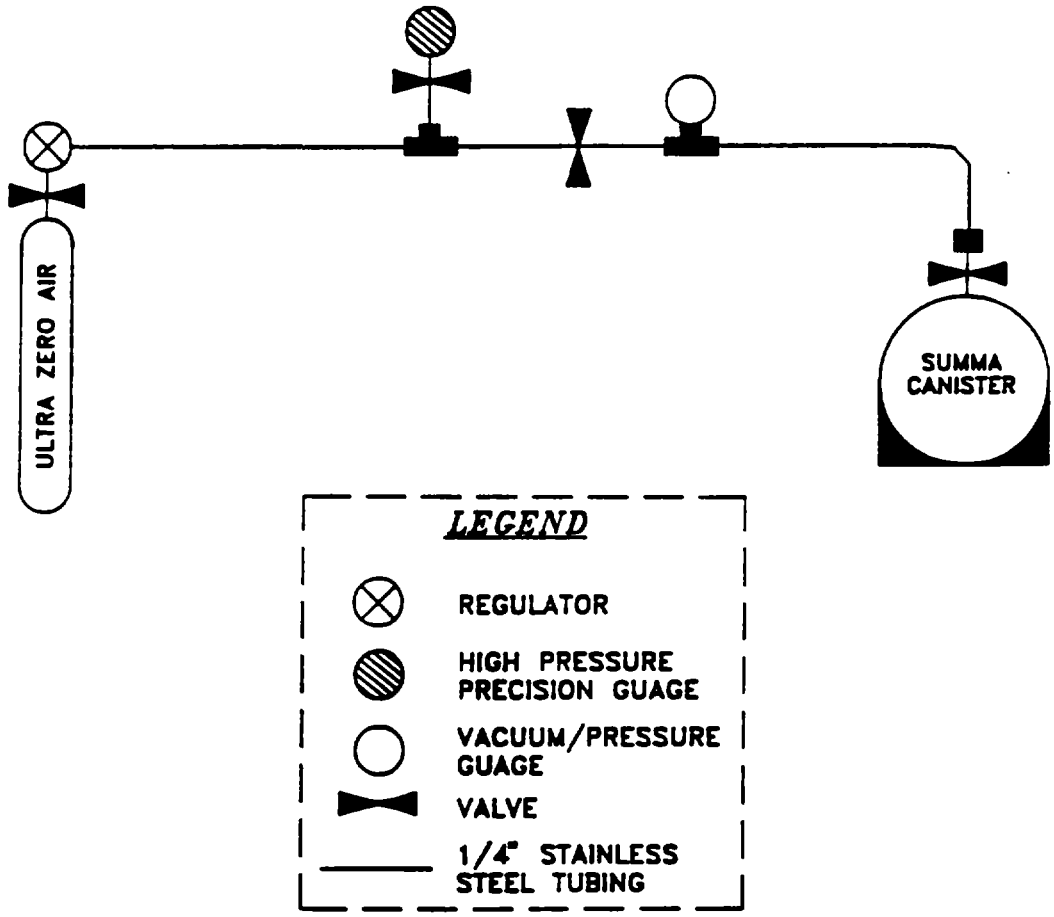


Figure 6: SUMMA Canister Analysis Train (Tekmar 5010 GC)

SOP #1705

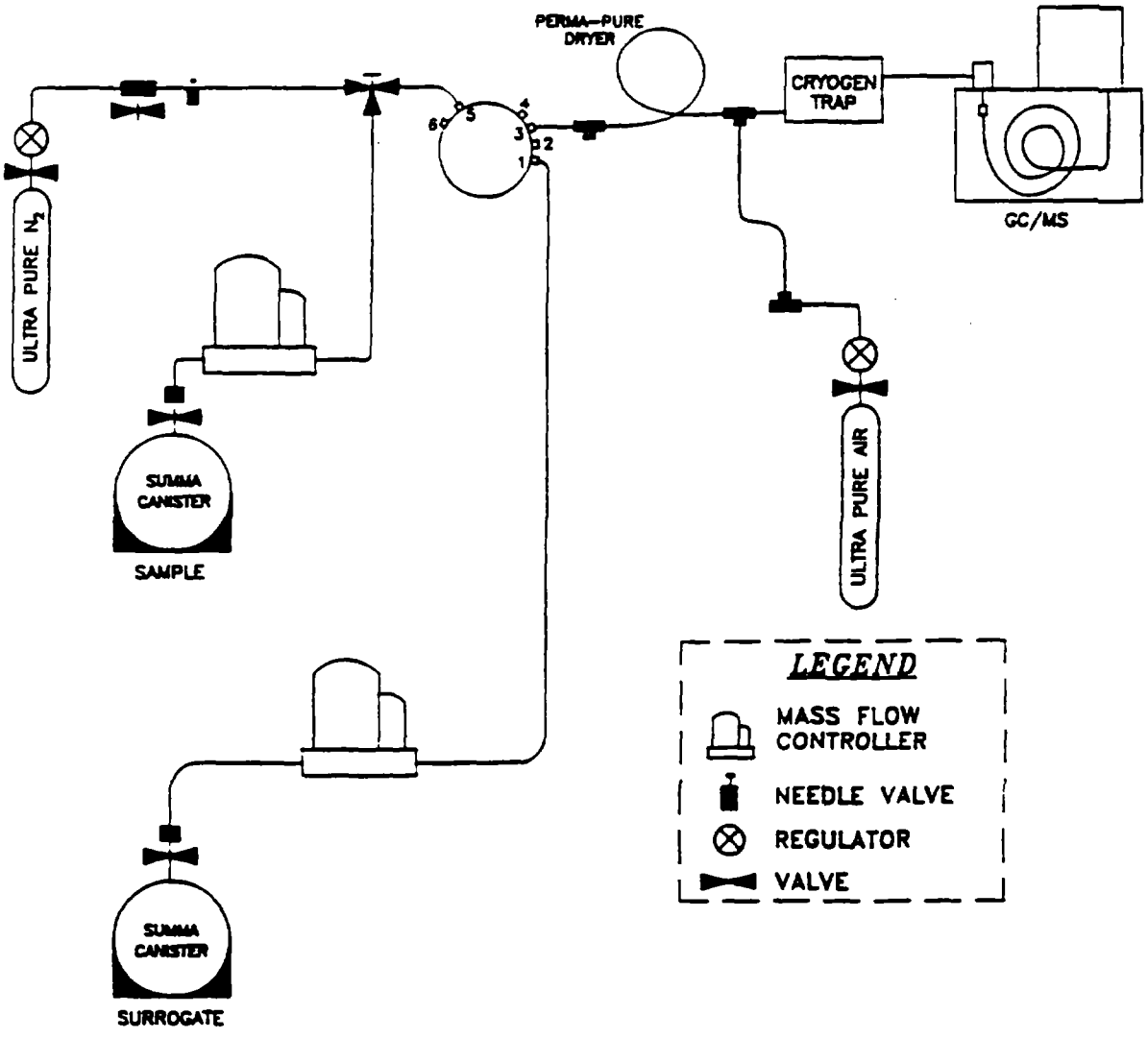


Figure 7: Canister Sample Absorbed onto Tenax

SOP #1705

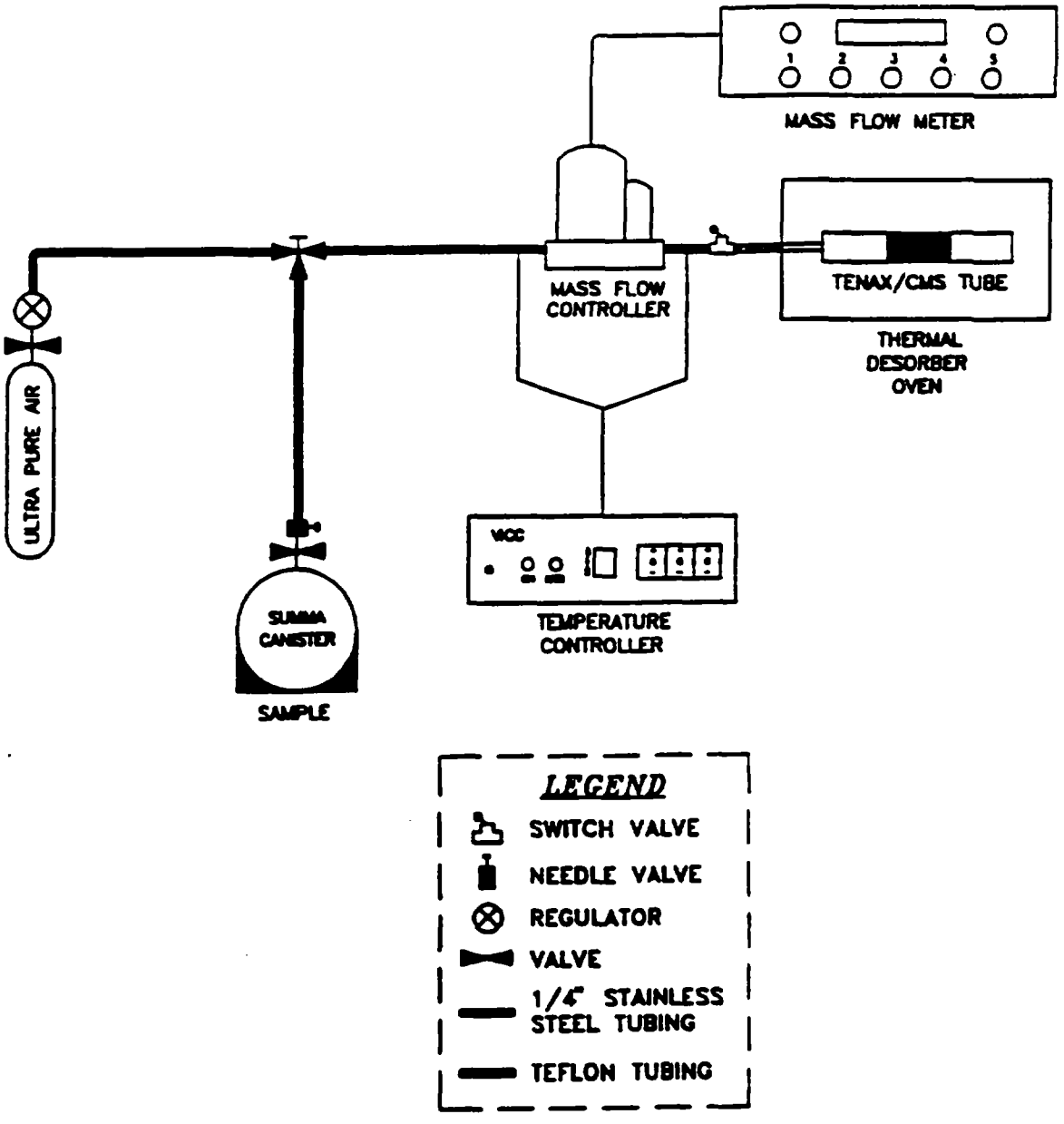


Figure 8: Teflon "Tee" Setup

SOP #1706

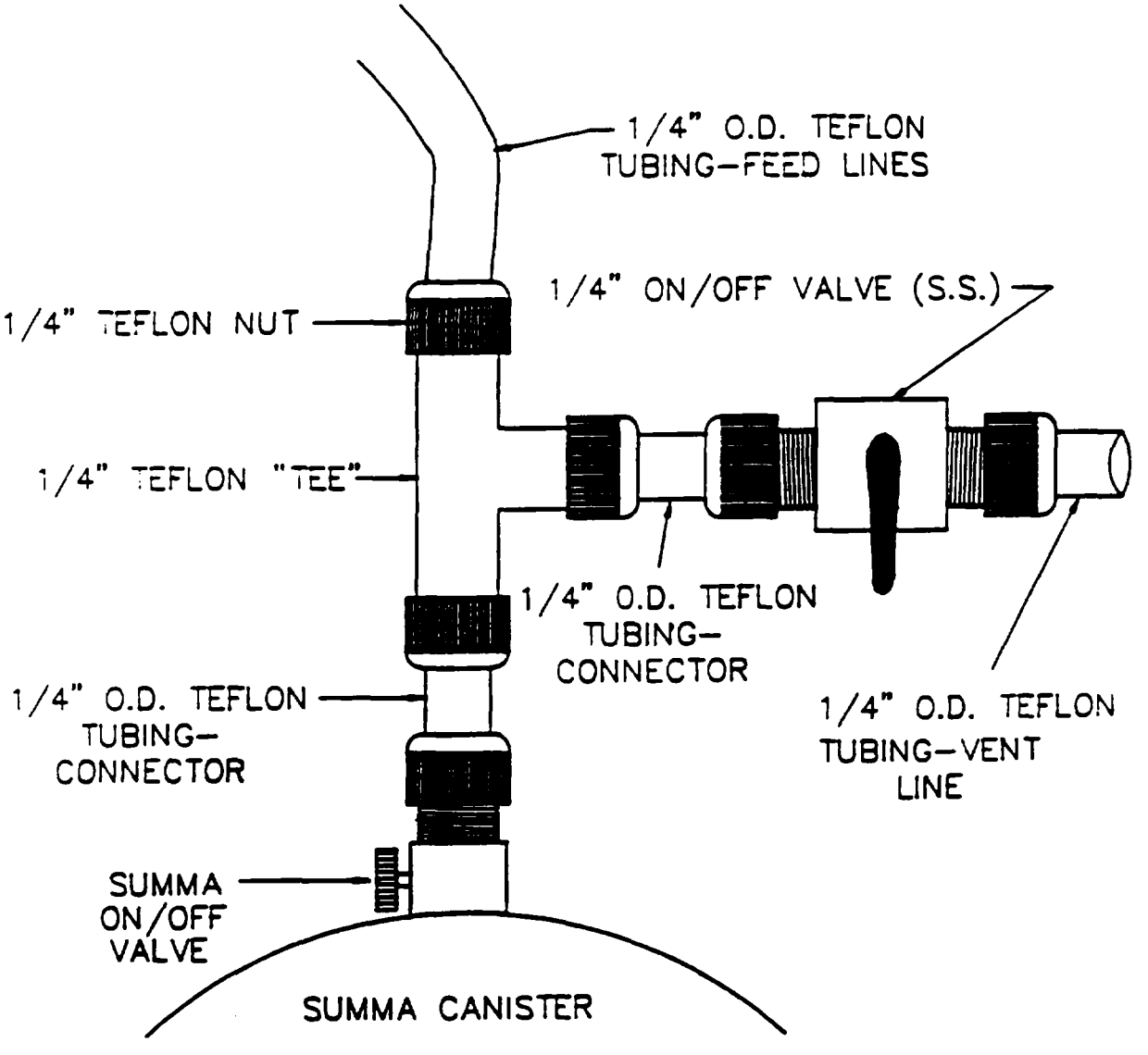


Figure 9: SUMMA Canister Charging System

SOP #1706

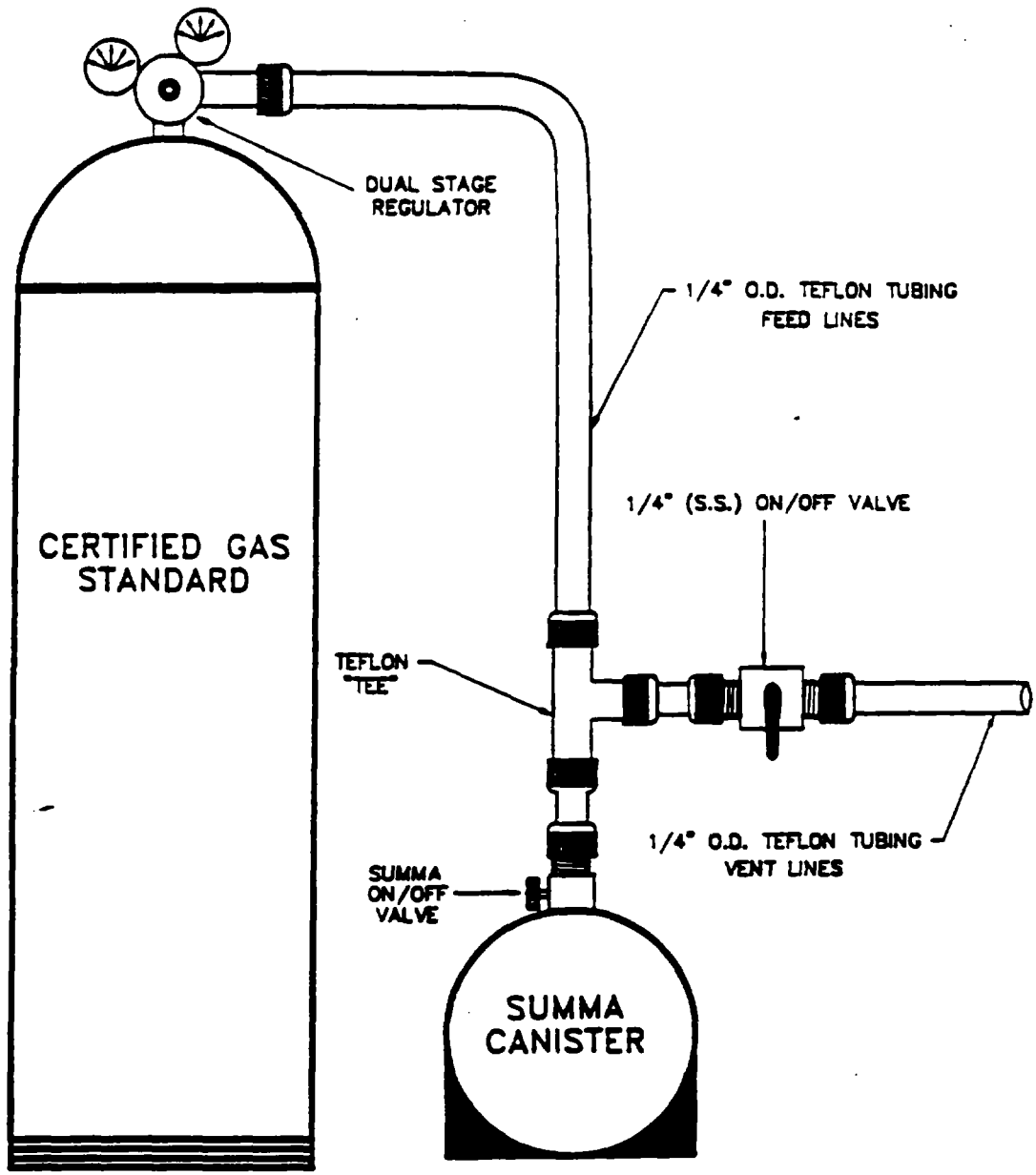


Figure 10: Septum "Tee" Setup

SOP #1706

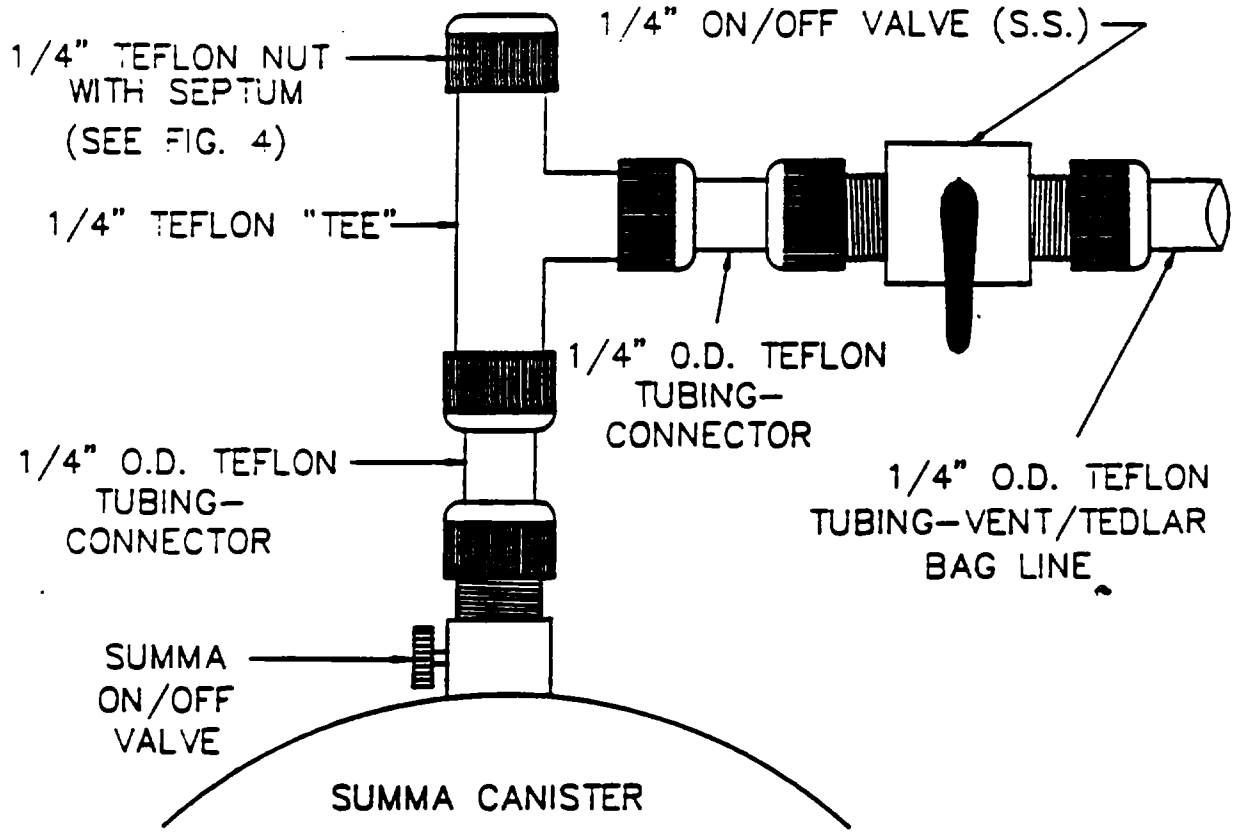


Figure 11: Teflon Nut With Septum

SOP #1706

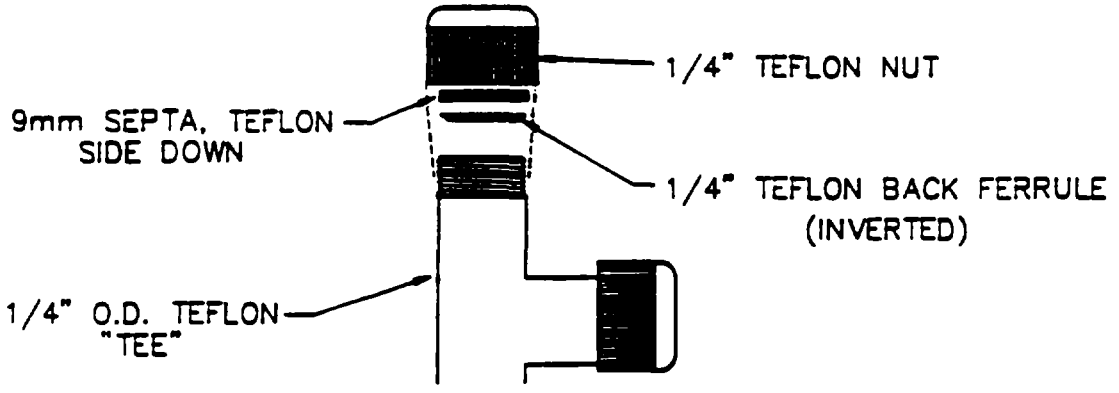


Figure 12: Phase Contrast Microscopy Filter Cassette

SOP #2015

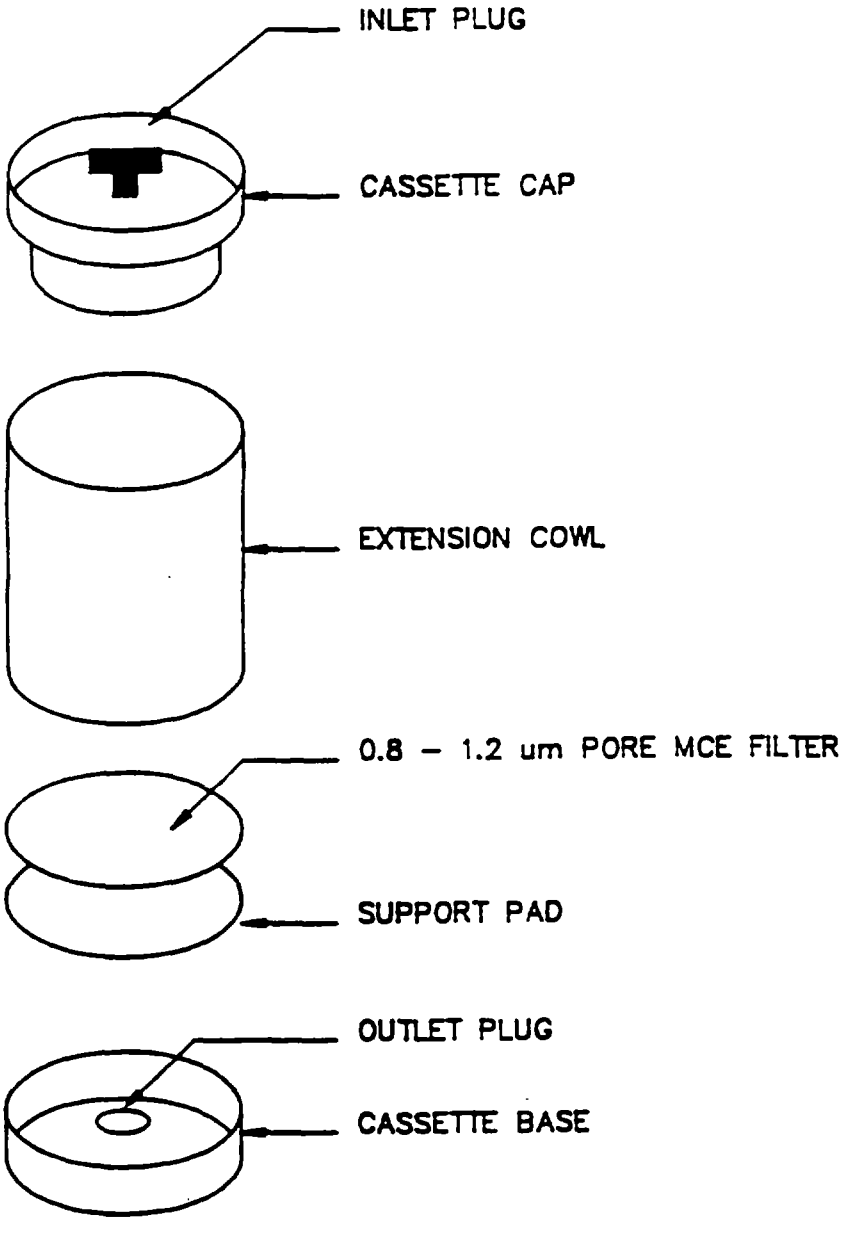


Figure 13: Transmission Electron Microscopy Filter Cassette

SOP #2015

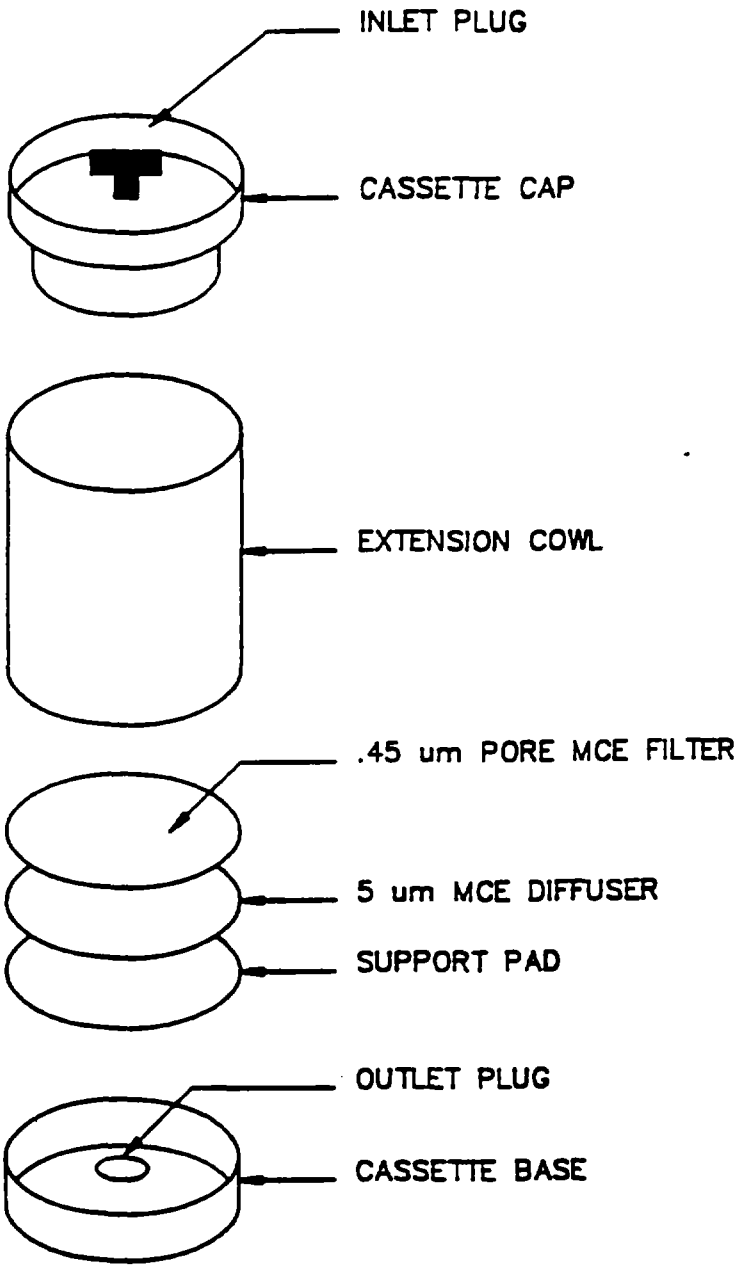


Figure 14: Personal Sampling Train for Asbestos

SOP #2015

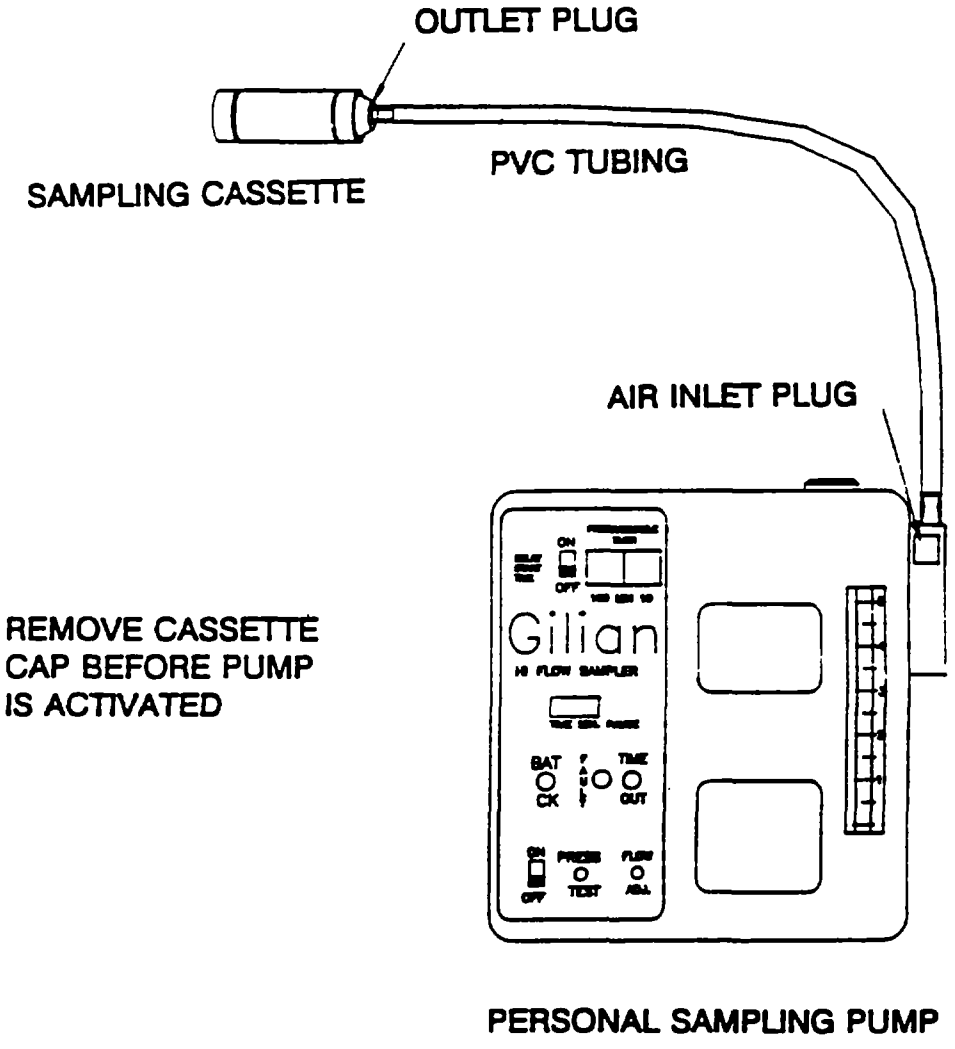


Figure 15: High Flow Sampling Train for Asbestos

SOP #2015

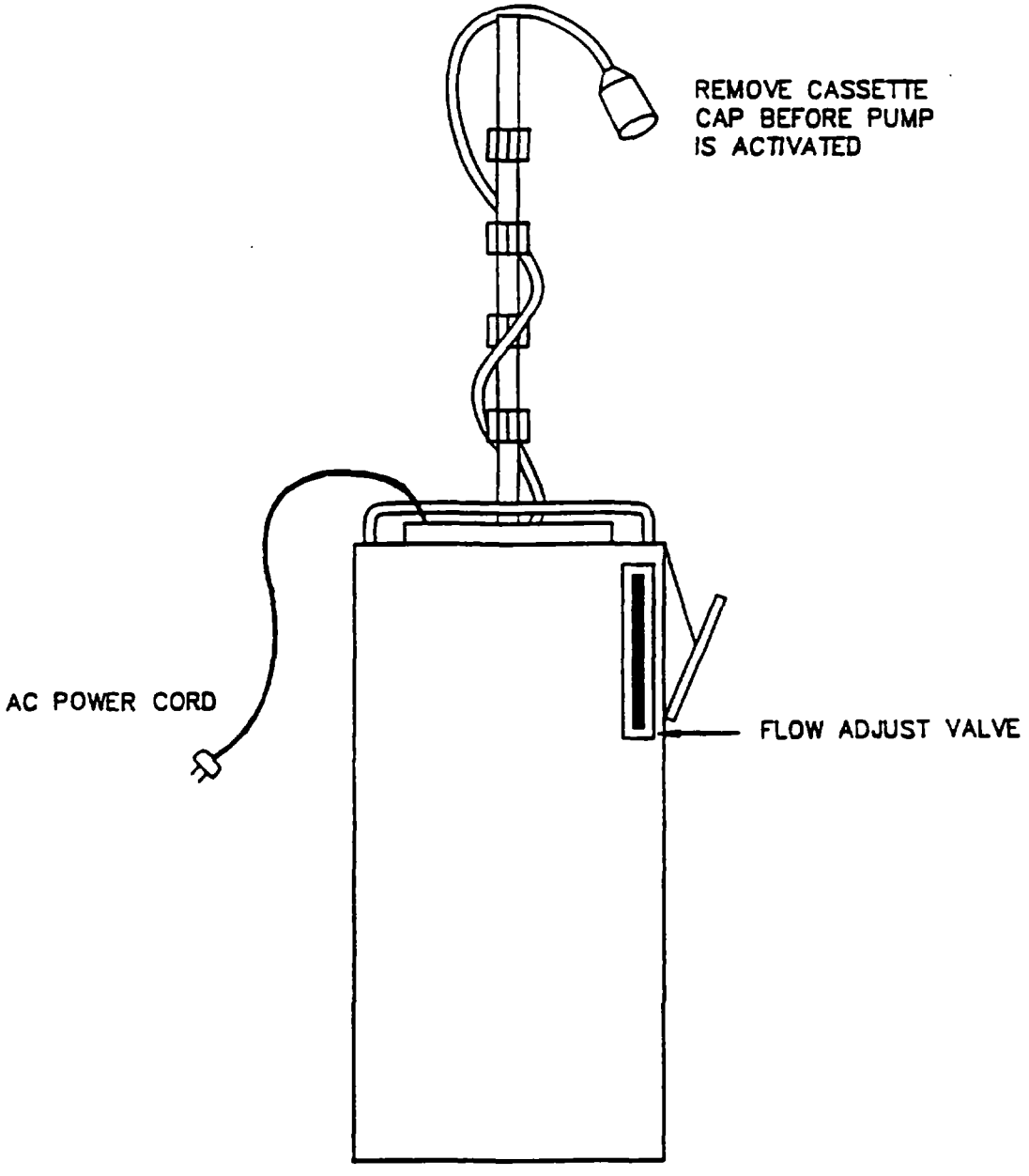


Figure 16: Calibrating a Personal Sampling Pump with a Bubble Meter

SOP #2015

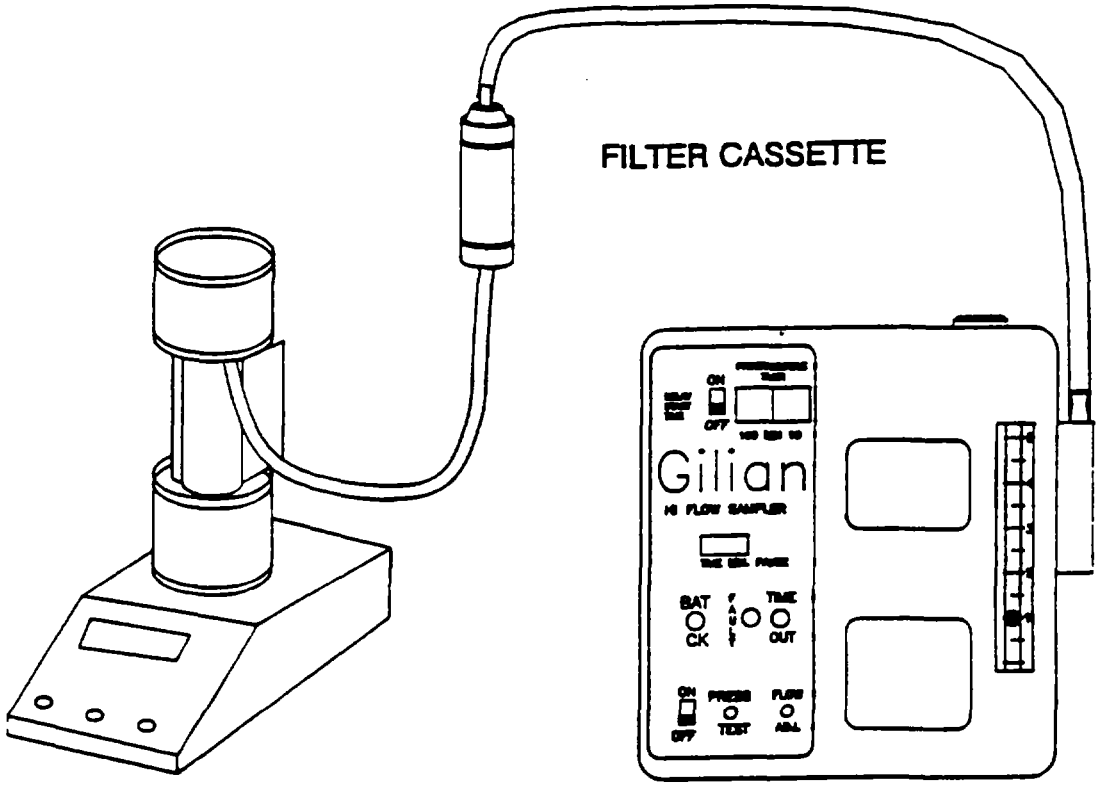


Figure 17: Calibrating a Rotameter with a Bubble Meter

SOP #2015

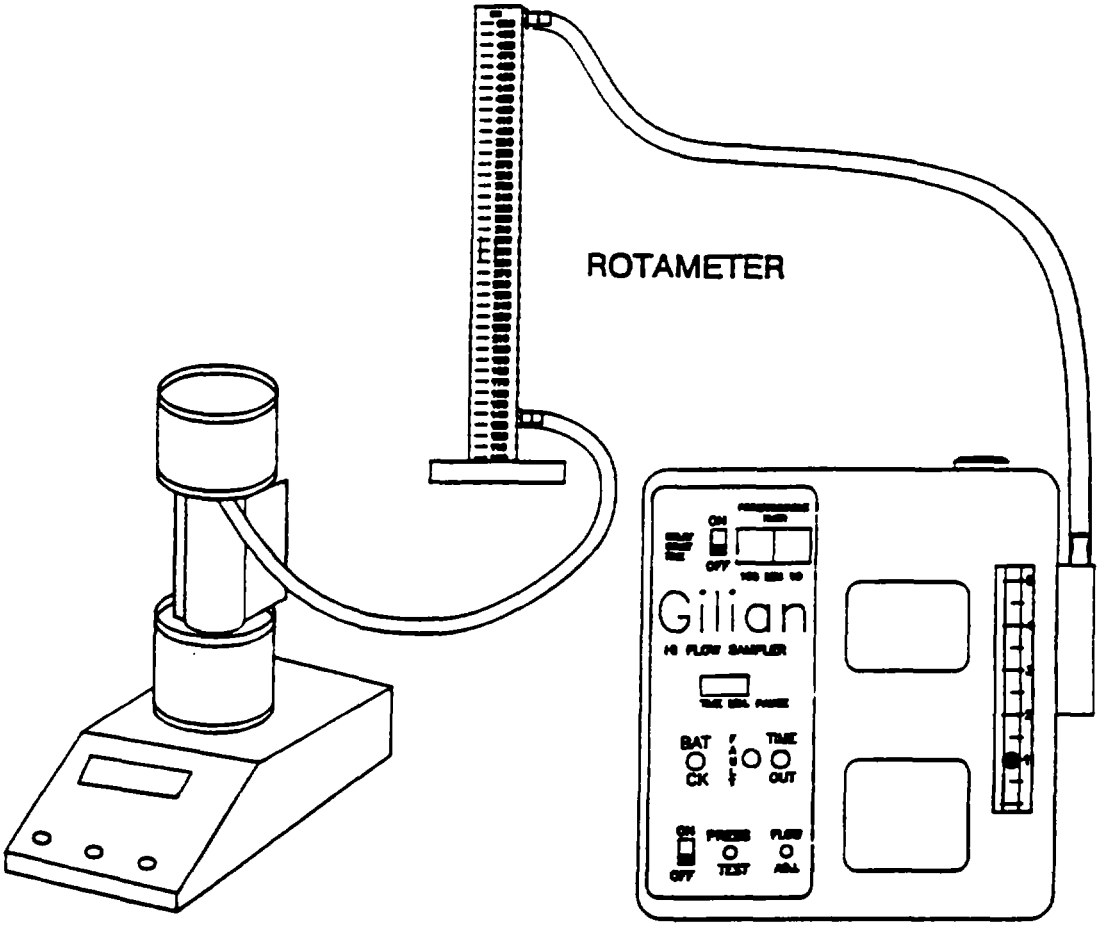


Figure 18: Calibrating a Personal Sampling Pump with a Rotameter

SOP #2015

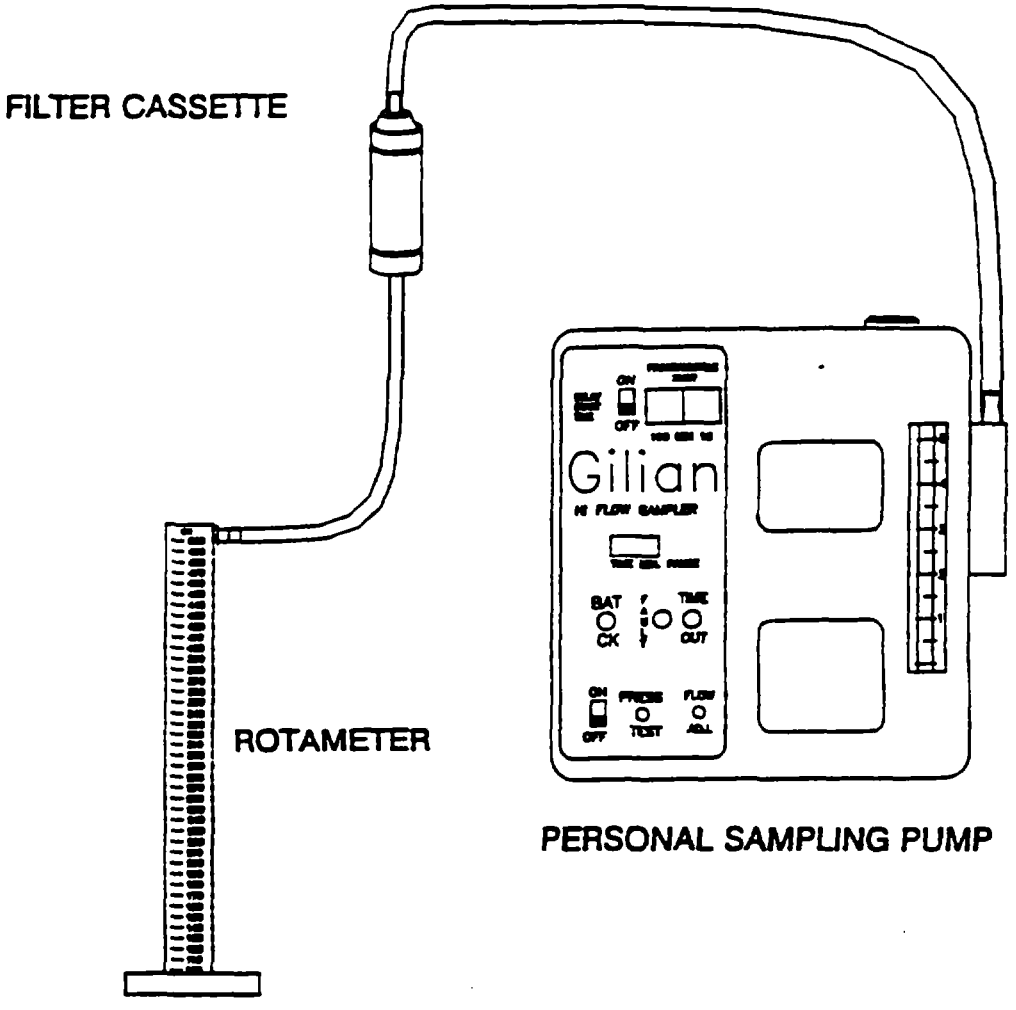


Figure 19: Tedlar Bag Sampling Apparatus

SOP #2050

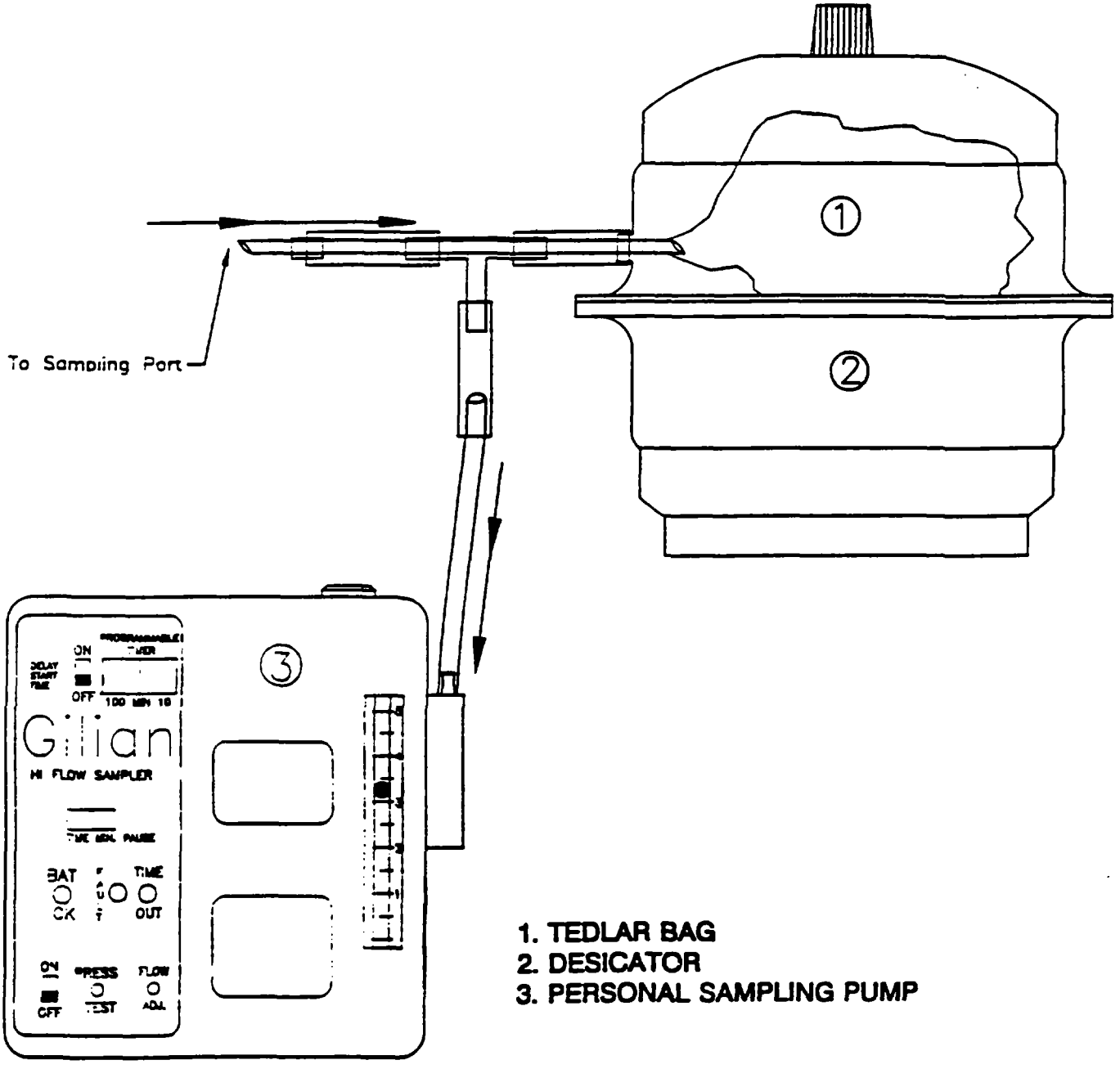
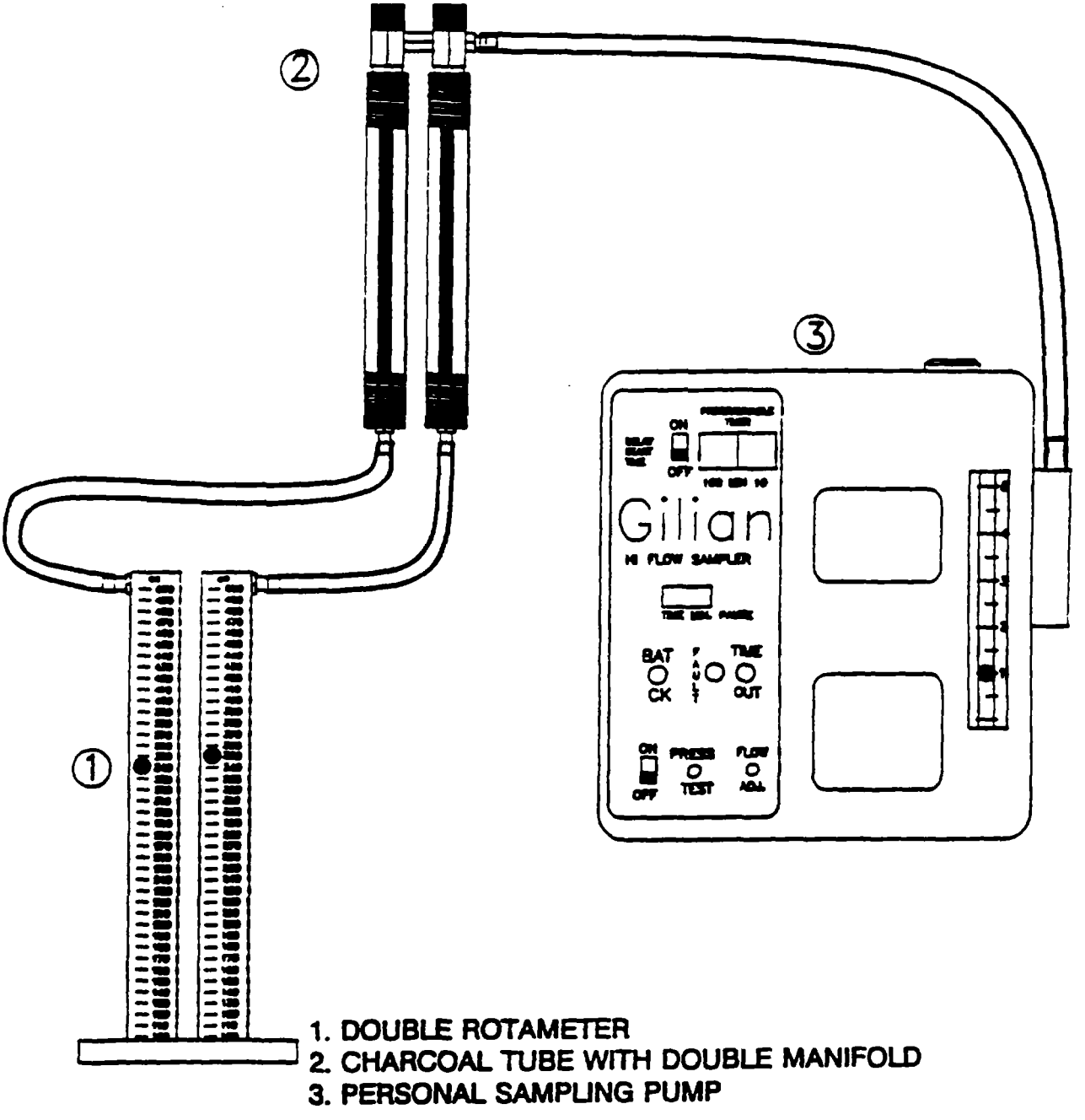


Figure 20: Calibrating a Double Manifold Charcoal Tube with a Rotameter

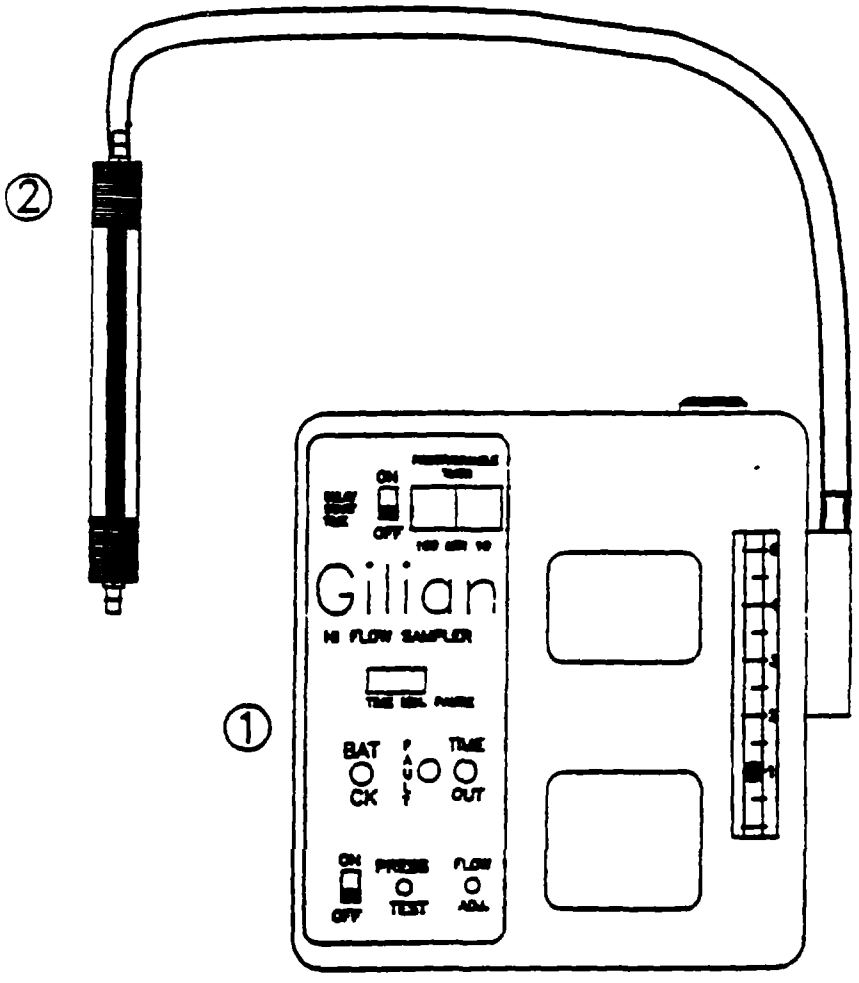
SOP #2051



1. DOUBLE ROTAMETER
2. CHARCOAL TUBE WITH DOUBLE MANIFOLD
3. PERSONAL SAMPLING PUMP

Figure 21: Charcoal Sampling, Straight

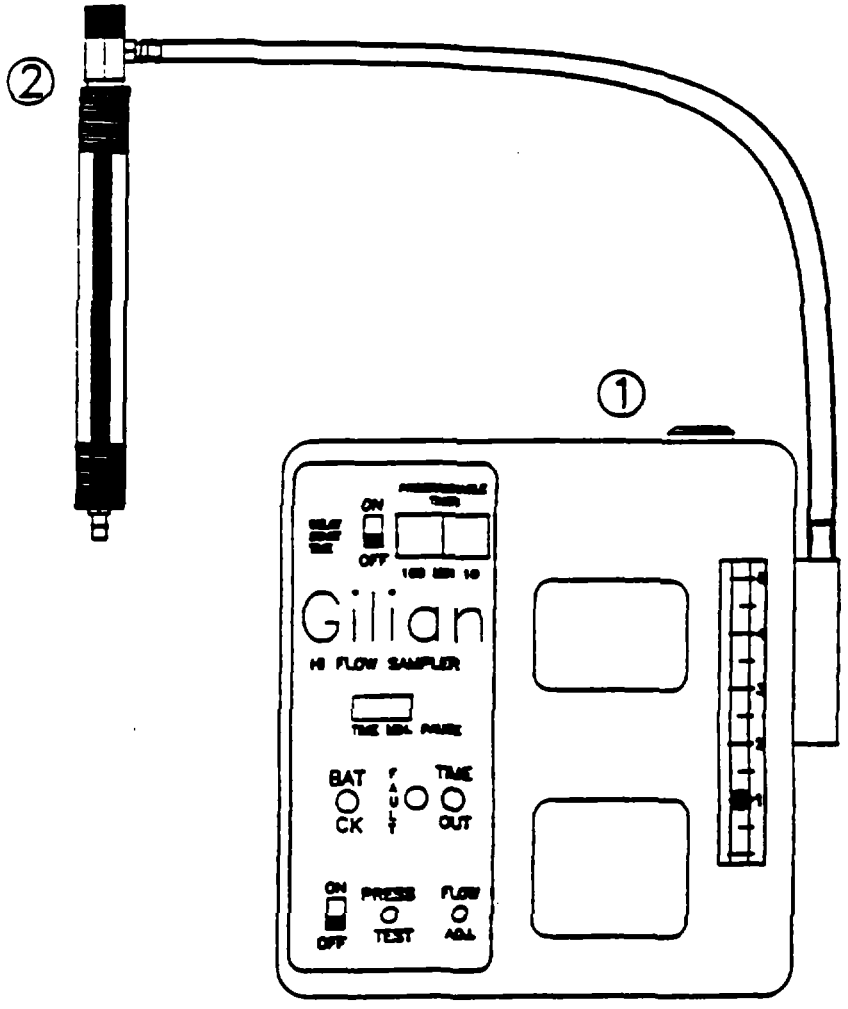
SOP #2051



1. PERSONAL SAMPLING PUMP
2. CHARCOAL TUBE - STRAIGHT

Figure 22: Carbon Sampling, Single Manifold

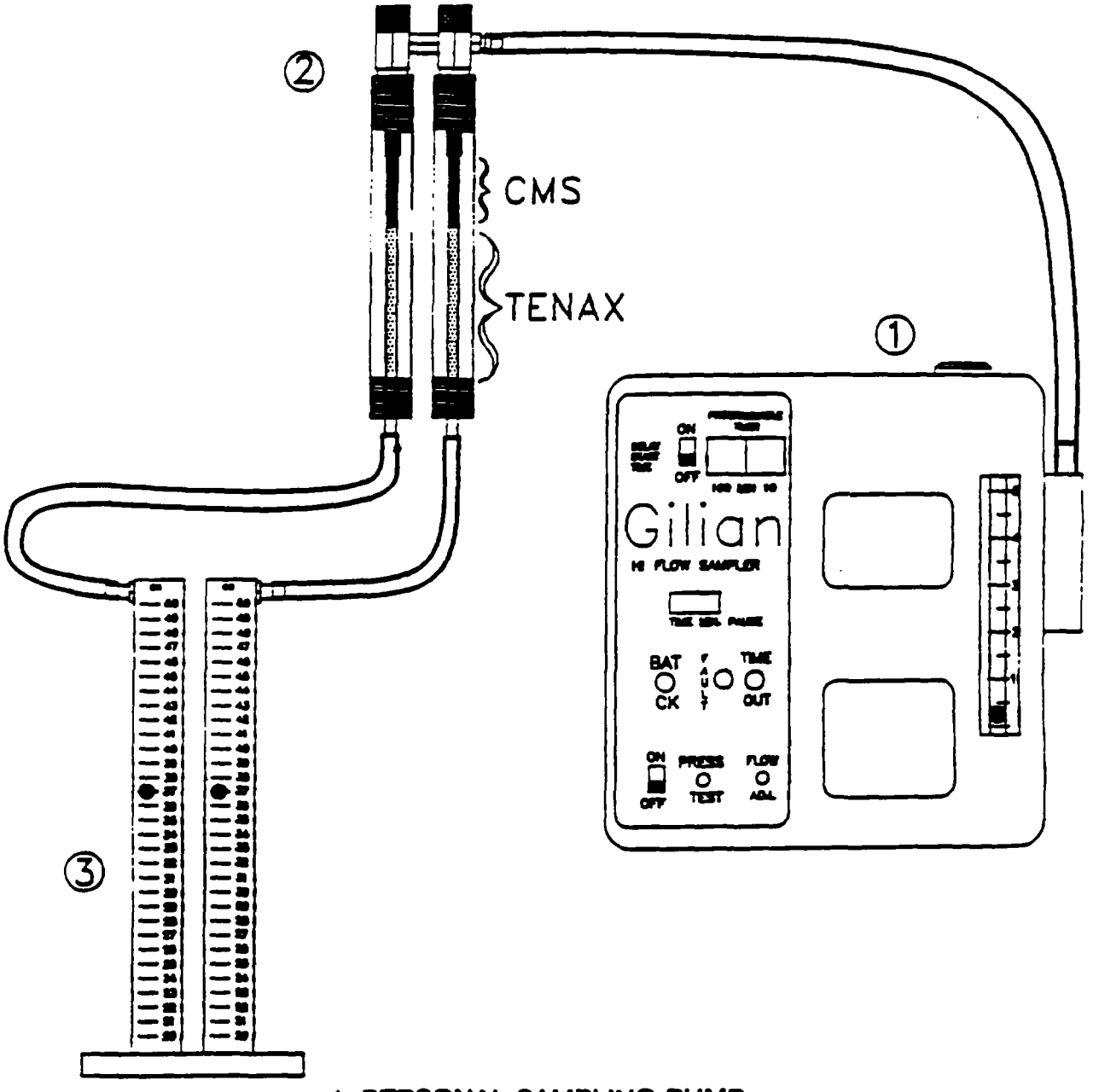
SOP #2051



1. PERSONAL SAMPLING PUMP
2. CHARCOAL TUBE SINGLE MANIFOLD (600mg or 150mg)

Figure 23: Tenax Calibration with a Secondary Calibrator

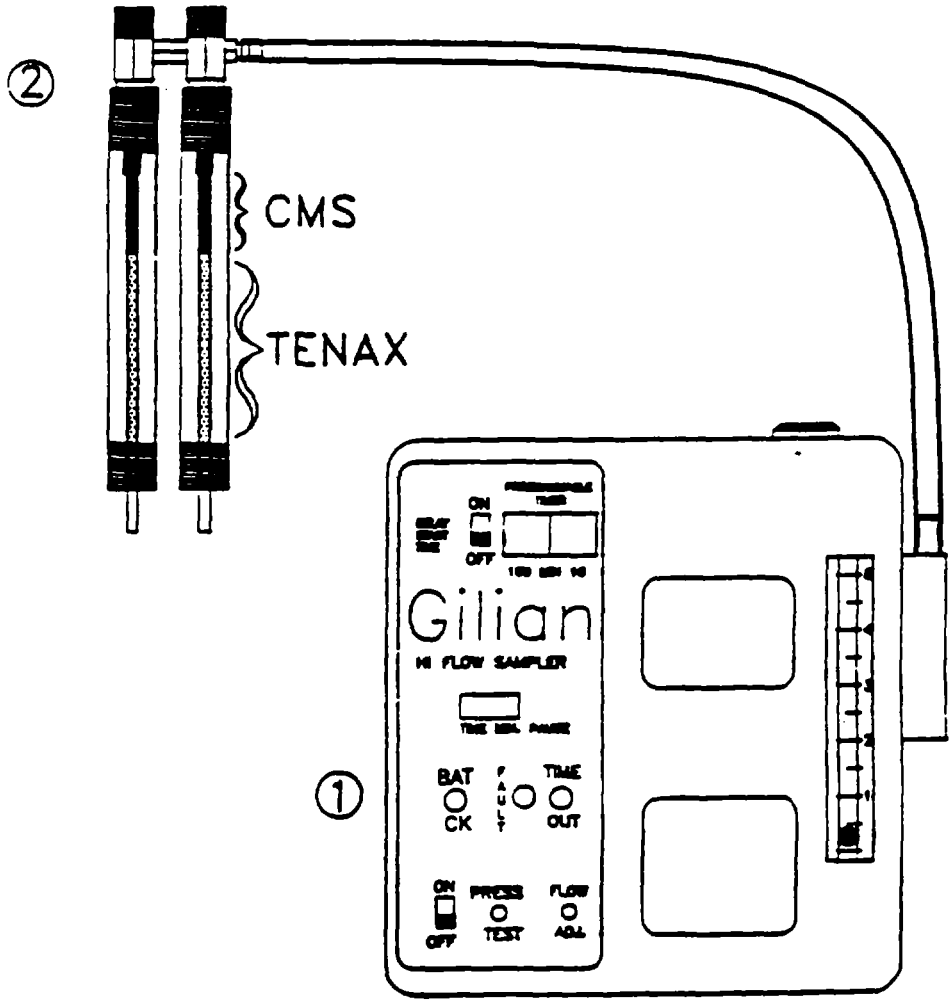
SOP #2052



1. PERSONAL SAMPLING PUMP
2. TENAX/CMS TUBE WITH DOUBLE MANIFOLD
3. DOUBLE ROTAMETER

Figure 24: Tenax/CMS Sampling Train

SOP #2052



1. PERSONAL SAMPLING PUMP
2. TENAX/CMS TUBE WITH DOUBLE MANIFOLD

Figure 25: Manometer

SOP #2069

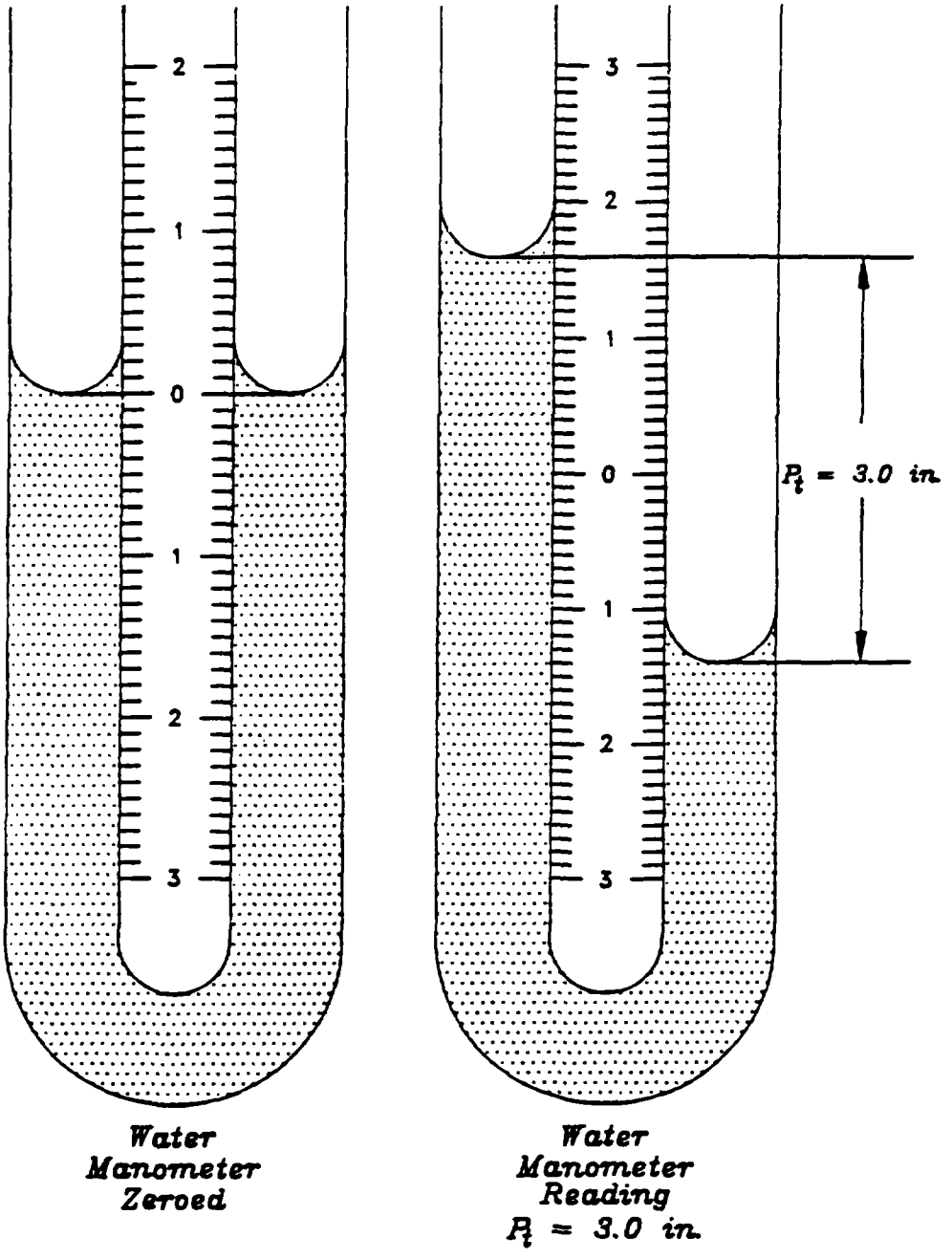


Figure 26: Canister Sampling Module

SOP #2069

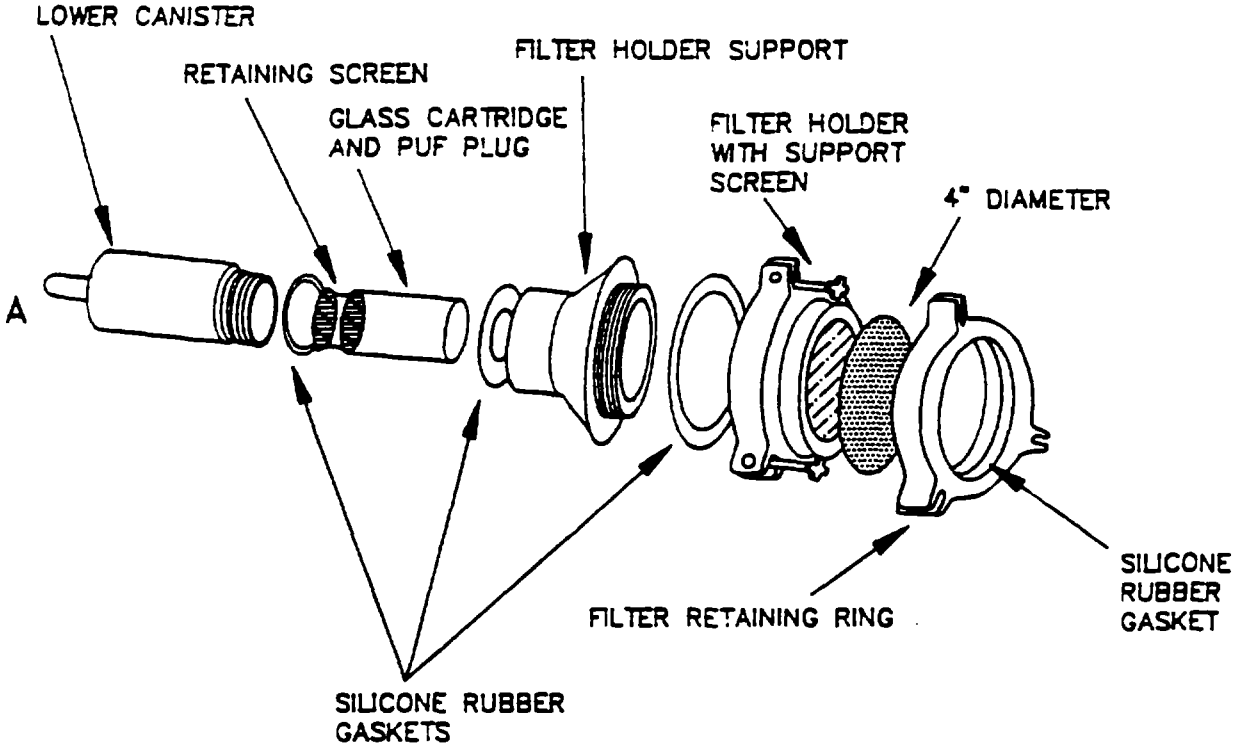
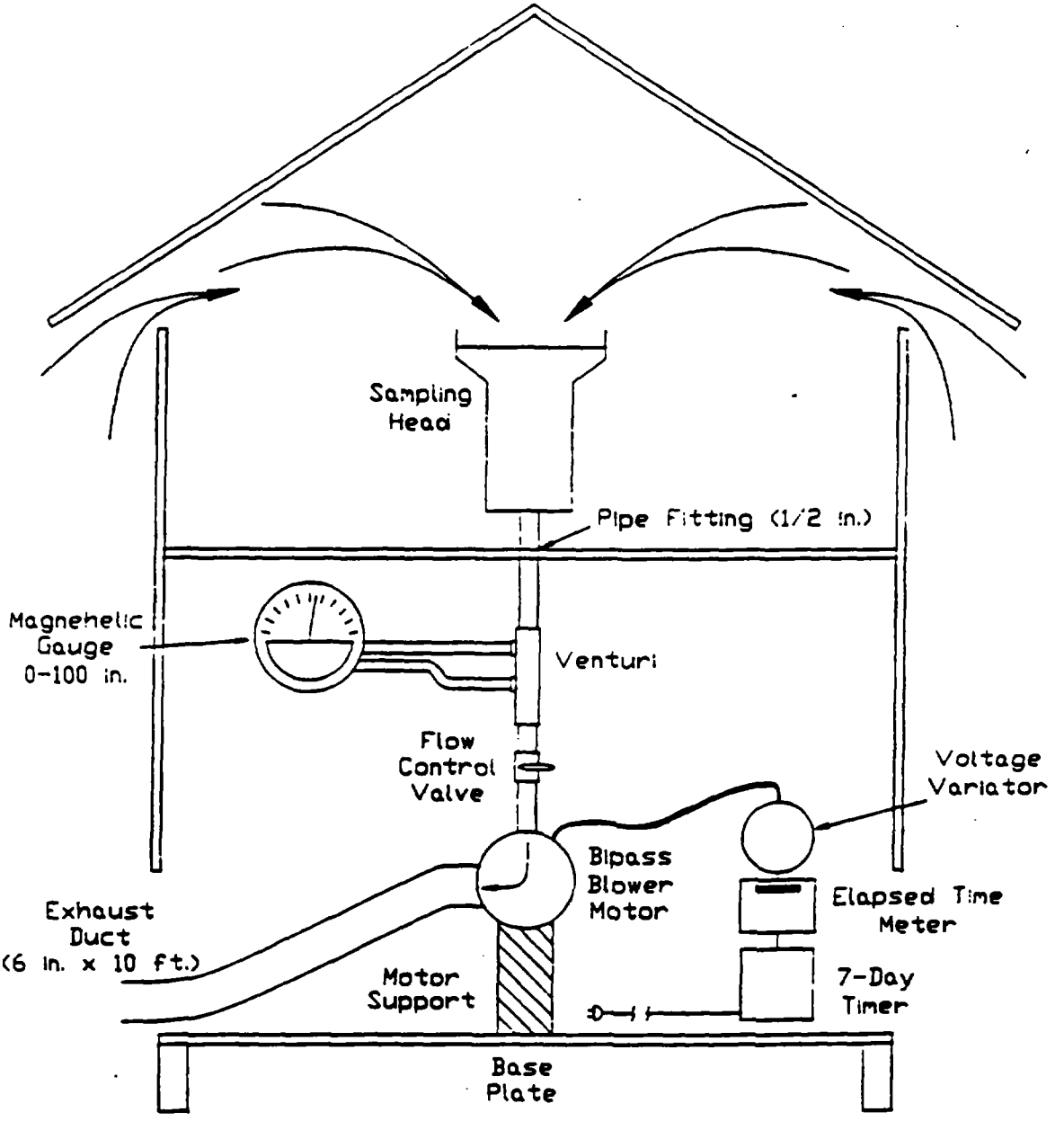


Figure 27: High Volume PUF Sampler

SOP #2069



APPENDIX B

Canister Sampling Field Data Sheet

Canister Sampling Field Data Sheet

SOP #1704

A. GENERAL INFORMATION	
SITE ID:	SHIPPING DATE:
SITE ADDRESS:	CANISTER SERIAL NO.:
	SAMPLER ID:
	OPERATOR:
SAMPLING DATE:	CANISTER LEAK CHECK DATE:

B. SAMPLING INFORMATION				
PARAMETER	START	STOP	MAXIMUM	MINIMUM
LOCAL TIME			NA	NA
ELAPSED TIME METER READING			NA	NA
INTERIOR TEMPERATURE				
AMBIENT TEMPERATURE				
CANISTER PRESSURE				
MANIFOLD FLOW RATE				
CANISTER FLOW RATE				
FLOW CONTROLLER READOUT			NA	NA
SAMPLING SYSTEM CERTIFICATION DATE:				
QUARTERLY RECERTIFICATION DATE:				

C. LABORATORY INFORMATION		
DATE RECEIVED:	INITIAL PRESSURE:	
RECEIVED BY:	FINAL PRESSURE:	
DILUTION FACTOR:		
INSTRUMENT	ANALYSIS DATE	ANALYSIS RESULT
GC-FID-ECD		
GC-MSD-SCAN		
GC-MSD-SIM		
ADDITIONAL RESULTS/COMMENTS:		
SIGNATURE/TITLE:		

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